

PATENT SPECIFICATION

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DRAWINGS ATTACHED

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(54) SEPARATION OF COMPONENTS

(71) We, ROSE, DOWNS & THOMPSON LIMITED, a British Company of Old Foundry, Cannon Street, Kingston-upon-Hull, Yorkshire do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the separation by selective crystallisation of one component or group of components from a mixture of components, such as the removal of the waxes from animal or vegetable oils, e.g. tallow and rice bran oil. The separation by selective crystallisation has been difficult to achieve on a commercial scale.

In accordance with the present invention, a process for removing an included organic or group of organic components from an extract from animal and/or vegetable raw material comprises mixing with the extract an organic solvent therefor, the temperature of the mixture being sufficiently high to cause the formation of a solution of the extract in the solvent, passing through the solution a liquid at a lower temperature to cool the solution and thereby to cause crystallisation of the component having the higher or highest temperature of crystallisation or the group of components having the higher or highest range of temperatures of crystallisation, removing the crystallised component or components, recovering the cooling liquid, which is immiscible or only partially miscible with the solution, and after cooling, recirculating the liquid. Subsequently the other component or components may be separated from the solvent; where there are more than two components or groups of components, a second component or group of components may be separated by a second crystallisation stage.

The cooled liquid is preferably water which is continuously circulated through the solution and which is continuously cooled. In that case, the solvent is one in which the components dissolved and with which water is immiscible or mixes to a limited extent. An example of such a solvent is methyl ethyl ketone.

The invention is applicable to the separation of a higher melting point fraction of a mixture of components from a lower melting point fraction; in this case the temperature of the solution is lowered to a value closely below the lower temperature of crystallisation of the components of the higher melting point fraction.

The invention will be more readily understood by way of example from the following description of a selective crystallisation process in accordance therewith and from the following description of one form of apparatus, for performing the process on a continuous basis, reference being made to the drawing accompanying the provisional specification which illustrates the apparatus.

In the selective crystallisation process, the mixture or organic components extracted from animal and/or vegetable matter, for example refined and deodorised palm oil is mixed with a solvent, such as methyl ethyl ketone or hexane, in which the components are soluble. The resulting mixture is heated to a temperature that is sufficiently high to cause the formation of a solution and that solution is passed into a process vessel where it is continuously but mildly agitated.

The solution is cooled by circulating water through the solution in the vessel, the water being sprayed in to the solution at the top of the vessel and, after passing through the solution, forming a pool at the bottom by virtue of its relatively high specific gravity. From the bottom of the vessel, the water containing a minor amount of the solvent is removed and pumped through refrigeration coils back to the top of the vessel.

The circulation of the cooled water through and in contact with the solution cools the vessel contents down to a temperature which is accurately determined by the cooling effected by the cooling coils. The distribution of the cooled water over the cross-section of the vessel ensures that the cooling is uniform. The temperature of the water is selected to cool the extract and solvent down to a temperature closely below the crystallisation temperature of the component or group of com-

ponents to be removed. Accordingly, the selected component or group of components are almost entirely crystallised and are separated from the liquid phase in any known manner.

- 5 If the original solution contains three or more components or groups of components additional to the solvent, the solution after crystallisation and removal of the first component or group, may be cooled further to
10 crystallise out the component or group with the second highest crystallisation temperature, and the selective crystallisation continued in stages in this way until only the solvent and one component or group of components remains. The final component or group may be
15 recovered by evaporation of the solvent or by other separation methods applicable to the materials involved.

- 20 Methyl ethyl ketone is slightly soluble in water and the circulated cooling water is therefore a solution of the solvent in water. The cooling liquid has four functions. It acts to cool the solution of the components in the solvent by direct contact, thereby achieving
25 more uniform cooling than if the vessel itself were to be cooled and obviating build up of solids on the vessel walls. Secondly, the presence of the water serves to keep the solution of the components in the solvent saturated with water. This keeps the crystallisa-
30 tion temperature of the component or components to be separated at a constant maximum value which minimises the load on the refrigeration system and makes it possible
35 to remove selectively and predictably the

higher melting components from the mixture by cooling the mixture to the appropriate temperature.

Thirdly, the freezing point of the water containing dissolved solvent is substantially
40 lower than that of pure water, thus enabling the refrigerating system to be operated at a temperature lower than the normal freezing point of water and allowing more rapid cooling of the oil solution to temperatures near
45 the freezing point of pure water.

Fourthly, because the water, or water containing solvent is a very poor solvent for the extract, the extract is not carried into the refrigerated heat exchanger in such quantities
50 as to cause deposition there resulting in rapid loss of cooling efficiency and eventual blocking of the heat exchanger.

EXAMPLE

One part by weight of refined and deodorised palm oil was mixed with two parts of
55 methyl ethyl ketone and the mixture heated to form a solution which was treated as described above, the temperature of the circulated water being controlled to bring the temperature of the solution to 14° C. The crys-
60 tallised high melting point fraction of the palm oil was separated by filtration and the remaining liquid low melting point fraction recovered by evaporation. Both fractions were
65 desolventised and dried by heating to 100° C. under vacuum. On analysis the original oil and the two fractions had the following characteristics:—

	Weight as percentage of original oil	Cloud point	Slip Point	Iodine value
Original Oil	100	26° C	38.4° C	52.4
High melting point fraction	23		53.2° C	35.4
75 Low melting point fraction	77	14° C		

The procedure was repeated using hexane as the solvent and giving the following results:—

	Weight as percentage of original oil	Cloud Point	Slip Point	Iodine value
Original oil	100	26° C	38.4° C	52.4
High melting point fraction	12		56.5	26.9
80 Low melting point fraction	88	19° C		

- 85 Apparatus for effecting selective crystallisation continuously is shown in the drawing and consists of a vertical column separated into a number of sections and having an agitator 12 driven by a variable speed agitator motor 13.

- 90 The mixture of components to be separated and the solvent-water-saturated methyl ethyl ketone or hexane—is passed through a heater 14 which raises the temperature to a value to give a complete solution. From the

heater 14, the solution passes through a cen-
95 tral pipe 15 to the lowermost section 16 of the column; as will be observed, the pipe 15 terminates part-way up the first section. A chilled solution of methyl ethyl ketone in water is continuously supplied from a cooler
100 17 to the second section 18 of the column, where it is sprayed into the column and descends as droplets, counter-current to the solution rising up the column. The water is removed at the bottom of the column and is
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formed by a pump 20 to the entry of the cooler 17.

A third section 21 of the column is provided as a de-entraining zone and is surmounted by a discharge section 22 from which the solution and crystallised material are continuously removed for subsequent separation in a filter, centrifuge, or the like.

Crystallisation of the higher fraction occurs in section 16 and the crystallised material, with entrained solution and water, pass upwardly into the de-entraining section 21. During the passage through section 21, and under the action of the mild agitation of the agitator 12, any agglomeration formation of the crystals is broken up, permitting the heavier component—the water—to become de-entrained and to descend to the bottom of the column for removal. Similarly, any crystals entrained with the water in section 16 are de-entrained in that portion of the section around the pipe 15 and are permitted to rise through the column for eventual removal.

WHAT WE CLAIM IS:—

1. A process for removing an organic component or group of organic components from an extract from animal or vegetable raw material, comprising mixing with the extract an organic solvent therefore, the temperature of the mixture being sufficiently high to cause the formation of a solution of the extract in the solvent passing through the solution a liquid at a lower temperature to cool the solution and thereby to cause crystallisation of the component having the higher or highest temperature of crystallisation or the group of components having the higher or highest range of temperatures of crystallisation, removing the crystallised component or components, recovering the cooling liquid which is immiscible or only partly miscible with the solution, and, after cooling, recirculating the liquid.

2. A process according to claim 1 in which the extract has at least three components or groups of components, and, after crystallisation of the component or group having the highest temperature or range of temperatures of crystallisation, the temperature of the solu-

tion is lowered to cause crystallisation of the component or group having the next highest temperature of crystallisation.

3. A process according to claim 1 or claim 2 in which after removal of the crystallised component or components, the solvent is recovered from the remaining solution.

4. A process according to any of the preceding claims in which the cooling liquid is water, the solvent selected being at most partially miscible therewith.

5. A process according to any of the preceding claims in which the solution is passed continuously through a vessel, counter-current to the movement of the cooling liquid.

6. A process according to any of the preceding claims in which the solvent is methyl ethyl ketone.

7. A process according to any of the preceding claims in which the extract is palm oil.

8. A process for separating a group of higher melting point organic components from an extract of animal or vegetable origin comprising adding to the extract, and dissolving the extract in, an organic solvent containing dissolved water, the solvent being only partially miscible with water, passing a solution of the solvent in water through the extract solution to saturate the solvent with water and to cool the solution to a temperature closely below the lowest temperature of crystallisation of the group to be separated, and thereby crystallising that group, removing the crystallised components, and recovering the solvent.

9. A separating process according to claim 8 in which the solvent is methyl ethyl ketone.

10. A process for removing an organic component from a group of such components, substantially as herein described and as illustrated in the drawing accompanying the provisional specification.

11. An organic material removed by the process of any of the preceding claims.

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10 Crystallisation of the higher fraction occurs in section 16 and the crystallised material, with entrained solution and water, pass upwardly into the de-entraining section 21. During the passage through section 21, and under the action of the mild agitation of the agitator 12, any agglomeration formation of the crystals is broken up, permitting the heavier component—the water—to become de-entrained and to descend to the bottom of the column for removal. Similarly, any crystals entrained with the water in section 16 are de-entrained in that portion of the section around the pipe 15 and are permitted to rise through the column for eventual removal.

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40 2. A process according to claim 1 in which the extract has at least three components or groups of components, and, after crystallisation of the component or group having the highest temperature or range of temperatures of crystallisation, the temperature of the solu-

tion is lowered to cause crystallisation of the component or group having the next highest temperature of crystallisation. 50

3. A process according to claim 1 or claim 2 in which after removal of the crystallised component or components, the solvent is recovered from the remaining solution. 55

4. A process according to any of the preceding claims in which the cooling liquid is water, the solvent selected being at most partially miscible therewith. 60

5. A process according to any of the preceding claims in which the solution is passed continuously through a vessel, counter-current to the movement of the cooling liquid.

6. A process according to any of the preceding claims in which the solvent is methyl ethyl ketone. 65

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8. A process for separating a group of higher melting point organic components from an extract of animal or vegetable origin comprising adding to the extract, and dissolving the extract in, an organic solvent containing dissolved water, the solvent being only partially miscible with water, passing a solution of the solvent in water through the extract solution to saturate the solvent with water and to cool the solution to a temperature closely below the lowest temperature of crystallisation of the group to be separated, and thereby crystallising that group, removing the crystallised components, and recovering the solvent. 75 80 85

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