

PATENT SPECIFICATION

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COMPLETE SPECIFICATION.

Process for the Removal of Water, Fats and Other Components from Material of Animal, Vegetable or Marine Origin.

We, JOHN CHARLES CAVANAGH and EDWIN GEORGE INMAN, Australian Citizens, both of 168 Adelaide Terrace, Perth, in the State of Western Australia, Commonwealth of Australia, 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 a process for the production of proteinaceous products by the removal of fats and flavour components from relatively dry proteinaceous raw materials of animal, vegetable or marine origin.

The process is applicable to the treatment of such materials as partially dried and/or partially de-fatted meat and fish materials or meat meals and fish meal; also various oil seeds, beans and grains. Such materials contain approximately 8% to 12% moisture, and, generally, not more than 20% moisture.

The end-solid-products of the process are highly refined solid proteinaceous materials, very substantially free from fat and flavour components, of good keeping qualities and suitable as foods for human beings, animals or birds. The fats and flavour components may be recovered as by-products.

It is already known to remove fat by counter-current extraction of relatively dry proteinaceous materials with various solvents. Such materials treated include meat meals and many kinds of oil seeds, beans etc. The solvents used are well-known fat solvents such as hydrocarbons and chlorinated hydrocarbons. (Generally speaking, non-water miscible solvents). The fat (or oil) leaves the counter-current treatment in solution in the solvent as an out-going

liquor commonly called miscella. The flavour components (i.e. taste and smell components) are not substantially removed by the solvent and they remain in the solid product from which the fat has been extracted. Thus is produced from relatively dry materials, a fat-free product but which still contains the flavour of the original material.

Our British Patent Specification No. 905,607 describes the removal of fat from wet or moist proteinaceous materials such as meat or fish in a multi-stage counter-current system with a solvent or mixture of solvents which is capable of mixing with water and also capable of dissolving fat and which has a boiling point below that of water. In this process the fresh solvent entering the last stage of the counter-current treatment (i.e. the stage from which the treated material passes out) dissolves fat from the material in that stage, which fat is progressively liberated or set free into a separate phase as the solvent progresses to the first stage (i.e. the stage into which the starting material enters) and becomes more and more mixed with the water entering with the raw material into the first stage. The combined liquor in the first stage exists in two separate phases, namely, solvent-water and fat. In this process, which treats wet starting materials, the flavour components are removed from the material, insofar as, during the treatment, they become dissolved in the mixture of solvent and water in the first stage and leave the counter-current system in the outgoing liquor from the first stage. Thus is produced from wet raw materials a fat-free product from which the flavour components have been removed.

In our co-pending application No.

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19953/65 (Serial No. 1,009,339) we have described a process in which the combined liquid of the first stage exists as a water-miscible solvent phase and a non-water-miscible solvent phase with fat dissolved therein.

However, it is not previously known to operate a counter-current system for the treatment of dry proteinaceous fatty materials with a solvent and in which the fat is removed and in which also flavour components are removed. The aim of the present invention is to achieve this very desirable object, namely, to treat dry proteinaceous materials in counter-current with a solvent and to remove both fat and flavour components.

The present invention resides in a process for the removal of fats and flavour components from dry proteinaceous materials of animal, vegetable or marine origin which comprises treating the comminuted material in counterflow with an organic solvent or mixture of solvents which is miscible with water and which is capable of dissolving fat and which has a boiling point below that of water and distilling the outgoing extraction liquor to recover the solvent, characterised in that water is added to the material before its treatment or during its treatment, to ensure that the outgoing extraction liquor from the counterflow treatment separates into at least two liquid phases, the one phase being substantially a mixture of solvent and water with flavour components dissolved therein and the other phase containing substantially all the fat.

The two-phase outgoing liquor from the treatment may be separated by gravity or by centrifuging to recover the fat and to leave behind the solvent-water phase with flavour components dissolved therein. The solvent-water may then be subjected to fractional distillation to recover the solvent for re-use, thus leaving behind the water with flavour components dissolved therein. If desired, the water may be evaporated and the flavour components recovered as a syrupy concentrate or as a dry powder.

Alternatively, the outgoing extraction liquor may, firstly, be distilled to recover the solvent, leaving behind the fat and also the water with flavour components dissolved therein. The fat is then separated by gravity or by centrifuging and the water may be evaporated to recover the flavour concentrate as a syrup or powder.

The selection of solvent may be made within wide limits, regard being had to the nature of the material to be treated, the ultimate use of the treated material and the costs involved. Acetone is a suitable solvent, as is a mixture of equal parts by volume of acetone, ethyl acetate and ethyl alcohol. This latter mixture is particularly

suitable for ensuring that substantially all the flavour components are removed from the material. It is considered preferable to avoid the use of cyclic hydrocarbons, chlorinated hydrocarbons or other solvents which may be objectionable from the viewpoint of any harmful effects of fumes in regard to the health of operating personnel or which may leave undesirable residues or taints in the end products of the process.

Depending upon the nature of the flavour components in the particular raw material and the extent to which it is desired to remove them, the choice of solvent may be varied. For example, using acetone, it is found that the flavour components may be fairly substantially removed in the counter-flow treatment, this taking place mainly in the early stages during which the acetone is mixed with water in proportions of the order 30% to 10% by volume but components not removed in the early stages may then still remain in the solid product during and after the latter stages, in which the water content of the acetone may diminish from about 10% down to about 2%. It may be found that acetone containing little or no water has little or no propensity for dissolving the flavour components.

On the other hand, a solvent such as an equal mixture by volume of acetone, ethyl alcohol and ethyl acetate is found to dissolve flavour components not only during the early stages when admixed with say 30% to 10% by volume of water, but also in the latter stages, when admixed with say 10% down to 2% or less by volume of water. A solvent of this kind is particularly valuable if it is desired to get very thorough extraction of flavour components.

The solvent is one which is substantially miscible with water and is also capable of carrying reasonable amounts of fat and water in one phase.

It is desirable to keep the amount of solvent to a minimum in order to keep solvent recovery costs down. For practical purposes the ratio of solvent in backflow to solid material in forward flow is of the order of $1\frac{1}{2}$ to $2\frac{1}{2}$ parts by weight of solvent to one part by weight of solid.

The degree of comminution of the material to be treated should be sufficient to enable the solvent to penetrate into the particles to achieve the desired degree of extraction of the fat or oil and/or other components.

In the case of animal or fish materials, it is found that most of the particles are of a porous, fibrous nature and are fairly readily penetrated by the solvent. However, it is found that some of the vegetable materials such as copra, peanuts and cocoa nibs are not readily penetrated by the solvent unless

the particle size is reduced to approximately 0.01 inch (approximately 0.25 mm.)

In cases where thorough extraction is desired, it appears that, generally speaking, and with most types of materials, the particle size should be reduced to approximately 0.01 inch (approximately 0.25 mm.)

It is also found that many of the raw materials which are of a fatty or oily nature are difficult to comminute to a small particle size. In these cases (e.g. copra, peanuts, cocoa nibs), it is found practicable to work with the feed material in a minced or flaked condition in the first or early stages of the counter-current system and to follow this with a secondary and finer comminution in the presence of the solvent, so that the latter stages of the countercurrent system are operated with a solid particle size approximately 0.01 inch (0.25 mm.).

The various features of the invention will be better understood by reference to the following specific examples.

EXAMPLE I.

Using as raw material shredded copra in a six-stage countercurrent treatment, the solvent being acetone. Each stage consisted of agitating the mixture of copra with the solvent liquor in a vessel for a period of approximately five minutes at temperature approximately 50°C, then filtering and squeezing the liquor from the solid matter and moving the solid matter forward to the subsequent stage vessel and passing the liquor backwards to the previous stage vessel. Each quantity of forward flow material entering the first stage was 30 grams of copra and to it was added 15 mls of water. Each quantity of acetone entering the last stage as backflow was approximately 45 mls of acetone in relation to each 30 grams of copra forward flow. The countercurrent system was brought into equilibrium conditions by rejecting the first several batches of outgoing solid product and of outgoing liquor. In the fourth stage, the mixture of solid matter and liquor was transferred for finer comminution to a disintegrator with high speed rotating cutter blades and then transferred back to the fourth stage vessel. (By explanation of the conditions existing during the treatment, it is to be noted that the 30 grams of copra entering as each batch of forward flow raw material contained approximately 8% water, i.e. a quantity of approximately 2.49 grams or 2.4 millilitres of water. To each batch was added 15 mls of water, making a total of 17.4 mls of water. Therefore, as the acetone solvent passed backwards from the sixth stage to the first stage, a quantity of 45 mls of acetone existed in the first stage in admixture with 17.4 mls of water, that is, in the proportions approximately 72 parts by

volume solvent to 28 parts by volume water. In such a mixture, fats and oils are substantially insoluble and will exist as a separate phase. However, the clean or fresh solvent entering the last or sixth stage was in contact with solid in the sixth stage which contained very little water (and likewise in the fifth stage), so that, in these stages, the solvent contained water of the order of only about 2% to 4%. Solvent containing 2% to 4% water will readily dissolve fat or oil, so that oil removed from the material in those stages was in true solution in the solvent. This oil, in true solution in the solvent in the sixth and fifth stages was thrown out of solution as the solvent progressed backwards into the first stage and became mixed with water to the amount of approximately 28% by volume).

The outgoing solid product from eight batches (equivalent to 240 grams of ingoing copra) was combined and, after removal of entrained solvent by evaporation, weighed approximately 53 grams. This product was milled into a fine powder, almost pure white and it had no detectable flavour of copra.

The outgoing extraction liquor from eight batches was combined and it consisted of two phases, the one being all or substantially all oil and the other being all or substantially all solvent-water with dissolved flavour components. The total volume of liquor was approximately 400 mls and to this was added a further 200 mls of water.

The solvent was removed from the two-phase liquor by fractional distillation, leaving behind a two-phase liquor consisting of oil and water, the latter with dissolved flavour components and a small amount of suspended solid matter.

The oil was separated by centrifuging and was then filtered. It weighed approximately 164 grams and was almost water-white and had the appearance and character of high grade coconut oil.

The aqueous phase containing the dissolved and suspended matter was concentrated by evaporation to yield a brown mixture of syrup and solid matter weighing approximately 21 grams and which had an intense flavour of coconut.

EXAMPLE II

In a manner similar to Example I above, a quantity of peanuts was so treated, the solvent being acetone. The peanuts were first roasted and the shells and red skins were removed and discarded. The peanut kernels were disintegrated to particle size of the order up to approximately 0.1 inch (2.5 mm). At the fourth stage of the countercurrent system, the solid matter was subjected to a finer comminution in the presence of the liquor. Similar phase con-

ditions existed throughout the system as in Example I.

From 240 grams of peanuts, the following products were obtained:—

- 5 (a) A quantity of approximately 105 grams of solid product, very pale yellow in colour and without any significant flavour of peanut.
- 10 (b) A quantity of approximately 117 grams of peanut oil of apparent high quality; and
- (c) A quantity of approximately 18 grams of a brown mixed syrup and solid matter with a taste of peanuts.

15 EXAMPLE III

A quantity of cocoa nibs was treated in a manner similar to Examples I and II above, except that in this case the solvent used was an equal mixture by volume of acetone, ethyl alcohol and ethyl acetate, previously purified by fractional distillation from a dilute aqueous solution of sodium hydroxide. The phase conditions were similar to the previous examples. The following products were obtained from 240 grams of cocoa nibs:—

- (a) Approximately 75 grams of solid product of pale brown colour and no significant flavour of cocoa.
- 30 (b) Approximately 125 grams of a fat, pale cream in colour, of the normal nature of cocoa butter; and
- (c) A mixed syrup and solid matter having a flavour of cocoa nibs.

35 EXAMPLE IV

The following illustrates the aspect as to whether or not the solvent itself or the mixture of solvent and water is responsible for dissolving flavour components:

40 If meat meal is agitated with warm acetone without addition of water, the flavour components are not substantially removed from the solid product. If the acetone is mixed with approximately 25% to 30% by volume of water, the mixture does substantially remove the flavour.

45 If, agitated with meat meal, the solvent is an equal mixture by volume of acetone, ethyl alcohol and ethyl acetate without addition of water, it will substantially remove the flavour. If this solvent is mixed with approximately 25% to 30% by volume of water, this mixture also will substantially remove the flavour.

50 With comminuted peanuts, the above conditions are also found to apply.

The application of the process of the present invention to the treatment of the materials specified above results in the following advantages:—

- (i) With certain materials, such as seeds which contain carbohydrate, the presence of water in the solvent causes swelling and assists in the penetration of the solvent into the material. 65
- (ii) The amount and nature of the flavour components removed by the solvent can be varied by an appropriate choice of solvent and/or by the presence of water in different proportions in the solvent. 70
- (iii) If desired, by an appropriate choice of solvent and/or by attaining an appropriate proportion of water in the solvent, certain proteins of a water-soluble nature may also be extracted along with the flavour components. 75
- (iv) By attaining a very thorough extraction of fat or oil and flavour components, the solid protein-containing product can be milled into a fine powder or flour of very good keeping characteristics. It appears that the fat or oil and the flavour components are relatively chemically unstable and are mainly responsible for spoilage of many natural or treated materials and, if they are thoroughly extracted, the solid matter has little or no further tendency to spoilage. As examples, the solid products from copra and peanuts appear to have keeping characteristics about similar to cornflour. 80
- (v) In the recovery of the fat or oil, the presence of water in the outgoing extraction liquor facilitates the total removal of the solvent from the fat or oil. For example, if the solvent is acetone and the outgoing liquor contains acetone and water in one phase and the fat or oil in a separate phase, when the acetone is removed by fractional distillation and by thereafter boiling off a very small amount of water, the fat or oil is virtually totally free of solvent. On the other hand, if water is not present and the fat or oil and solvent are in the one phase, it is much more difficult to remove the last of the solvent from the fat or oil, involving higher temperature and/or reduced pressure or a longer time and agitation. 85
- (vi) Many natural or treated materials containing protein or mixtures of protein with other solid components such as carbohydrates possess oils and/or flavours objectionable or not needed or in too great propor- 90

tion for the diets of particular animals or human beings. It is considered to be of great value that the protein and associated solid nutritious products can be produced free from the particular oil and/or flavour and thus be available for incorporation or admixture in any kind of diet needed or desired by the particular animal or human being.

The examples given herein illustrate the basic principles of the invention. For carrying out the invention for the treatment of large amounts of materials on a commercial scale suitable apparatus such as is described in our co-pending British Patent Application No. 29958/60 (Serial No. 960,369) may be used. For the finer comminution in the presence of the solvent any suitable machine, such as a Jordan refiner as used in the paper industry, may be used.

The term "fat" or "oil" used herein is intended to cover materials commonly known as fats which are solid at normal temperatures and those commonly known as oils which are liquid at normal temperatures.

WHAT WE CLAIM IS:—

1. A process for the removal of water, fats and other components from relatively dry proteinaceous materials of animal, vegetable or marine origin which comprises treating the comminuted material in counter-

flow with an organic solvent or mixture of solvents which is miscible with water, which is capable of dissolving fat and which has a boiling point below that of water and distilling the outgoing extraction liquor from the counter-flow treatment to recover the solvent, characterised in that water is added to the material before its treatment or during its treatment to ensure that the outgoing extraction liquor from the counter-flow treatment separates into at least two liquid phases, the one phase being substantially a mixture of solvent and water with flavour components dissolved therein and the other phase containing substantially all the fat.

2. A process as claimed in Claim 1 wherein the solvent is acetone.

3. A process as claimed in Claim 1 wherein the solvent is a mixture of equal parts by volume of acetone, ethyl acetate and ethyl alcohol.

4. A process as claimed in any one of the preceding claims wherein the material is subjected to a secondary comminution during the counterflow treatment.

5. A process for the removal of water, fats and other components from relatively dry proteinaceous materials of animal, vegetable or marine origin substantially as herein described.

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