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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 removal of fats and water from materials of animal, vegetable or marine origin.

The process is applicable to the treatment of such materials as meat, fish, whales and the scraps or waste from such materials; also, various oil seeds, beans and grains.

In the operation of the processes described in our British Patent Specifications Nos. 905,607 and 41183/61 (Serial No. 1,009,338) fats and water are removed from protein-containing materials by treatment in a counter-current system with one or a mixture of organic solvents. The solvent or the mixture of solvents is capable of mixing with water and is able to dissolve fat and has a boiling point below that of water. The solid end-product from the treatment contains very little fat or water, but contains absorbed solvent. When the solvent is evaporated, the solid end-product is thus recovered, free from solvent and also substantially free from the fat and the water originally in the protein-containing material.

It is a feature of the processes described in the forementioned specifications that the solvent moving in the opposite or counter-current direction to the protein-containing material dissolves fat from the material in the last stage (i.e. the stage where the solvent enters the system and where the solid end-product passes out), which fat dissolved in

the solvent is liberated or set free into a separate phase as the solvent passes progressively towards the first stage i.e. the stage where the protein-containing material enters the system and from which the solvent passes out) and becomes mixed with the water contained in the ingoing protein-containing material. In our co-pending application No. 41183/61 (Serial No. 1,009,338) sufficient water is included in the solvent to ensure that the outgoing extraction liquor separates into at least two liquid phases. The outgoing liquid is a combined liquid of two phases, one phase being substantially a mixture of solvent and water and the other phase containing substantially all the fat.

The fore-mentioned processes also remove flavour components from the protein-containing material. The flavour components pass out of the treatment system dissolved in the mixture of solvent and water.

The solvent or solvents used in the fore-mentioned processes are basically of a type which are miscible with water and are also able to dissolve fat, but they are not able to mix with substantial water and also dissolve substantial fat at one and the same time. When the solvent is mixed with little or no water, it is able to dissolve fat. But, when mixed with substantial water, the solvent is not able to dissolve substantial fat and fat which is present in the solvent exists in a separate phase.

It has been found that the processes described in the fore-mentioned specifications present a physical difficulty in the separation of the combined liquid from the solid protein-containing material in the first stage, that is, at the point where the solid passes forwards into the second stage and the combined liquid passes out of the overall

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counter-current treatment system. If the temperature is low, such that the fat is in a solid condition and if it is in a large amount, separation is very difficult or almost impossible. The difficulty is lessened by operating at an elevated temperature such that the fat is in a liquid condition, but, even so, the fat is very viscous and separation may still cause some difficulty.

The processes described in the fore-mentioned specifications also have a restriction in their application, because the water-miscible solvents, when used at the elevated temperature and in a suitable amount in relation to the amount of protein-containing material, will generally dissolve the flavour components in the mixture of solvent and water which exists in the first stage and which passes out of the overall treatment system as the outgoing combined liquid. Hence, the processes are restricted in that they generally produce a solid end-product from which the flavour components are removed and this might not always be desired.

The present invention resides in a process for the production of a substantially defatted and de-watered proteinaceous end-product from a protein-containing material of animal, vegetable or marine origin and which contains fat and water, in which process is used a mixture of organic solvents containing one or more solvents substantially miscible with water and one or more solvents substantially non-miscible with water but capable of dissolving fat, the mixture of solvents having a boiling point or boiling range which lies below the boiling point of water, the protein-containing material being treated by the solvent mixture in a preferably continuous multi-stage counter-current process, of which the first stage is the one where the protein-containing material enters and from which the solvent mixture passes out and the last stage is the one where the new or clean solvent mixture enters and the proteinaceous end-product passes out and that the solvent mixture entering the last stage and passing towards the first stage progressively removes fat and water from the protein-containing material as it passes from the first stage to the last stage, characterised in that the combined liquid in the first stage exists in two separate phases of which the one phase consists substantially of water-miscible solvent or solvents and water and the other phase consists substantially of non-water-miscible solvent or solvents with fat dissolved therein. Thus, during the operation of the counter-current system, the fat which is dissolved from the protein-containing material in the last stage is not necessarily always set free or thrown out of solution into a separate phase as the solvent becomes mixed with water in the first stage of the process, but it remains dissolved

in the non-water miscible component of the solvent. Hence, in the first stage of the counter-current process, the combined liquid consists of two phases, the one phase consisting substantially of the water in the protein-containing material in admixture with the water-miscible components of the solvent and the other phase consisting of fat dissolving in the non-water miscible components of the solvent.

Because fat dissolved in the non-water miscible solvent is in the form of a thin or mobile liquid and not in the form of solid fat or a very viscous melted fat, the difficulty is overcome which occurs in the separation of the combined liquid from the protein-containing material in the first stage of the counter-current process. Furthermore, because solid fat and liquid fat will both dissolve in the non-water miscible components of the solvent, the process can be operated at any desired temperature either low or elevated or below or above the melting point of the fat in the protein-containing material.

If the protein-containing starting material is in a relatively dry condition (such as a moisture content of 20% or less), it is sometimes desirable to add water. The amount of water added depends on various circumstances. An amount such as approximately 50% of the weight of the dry material is suitable in some instances. The water is preferably added to the starting material or to the mixture of material and solvents in or near the first stage of treatment. The addition of water may be desirable in the case of dry materials when it is required to remove the taste and small components such as are soluble in mixtures of water-miscible solvent and water.

By using a suitable solvent mixture in accordance with the present invention it can also be arranged that the flavour components of the protein-containing material are not removed by the solvent, but that they remain in the solid end-product of the process. This may be accomplished by a choice of solvent mixture consisting mainly of non-water miscible components and with only a small or lesser quantity of water-miscible components and by operating at a suitably low temperature. In this way, the combined liquid in the first stage consists of a small amount of water-miscible solvent admixed with the water in the protein-containing material and at a low temperature which does not dissolve the flavour components. Thus, the flavour components remain in the protein-containing material and stay in the solid end-product of the process.

It has been found that a suitable solvent for this aspect of the invention may be prepared such as by using a petroleum hydrocarbon solvent and blending with it water-miscible solvents which will normally pene-

trate moist materials (such as acetone and ethyl alcohol) together with another desirable solvent, such as ethyl acetate. The choice of solvent mixture is governed by, firstly, that it must achieve the essential object of penetrating into the moist material to dissolve the fat (or oil) and to liberate the water in a separate phase and, then, by other considerations such as to be recoverable by normal distillation practices for re-use and not to leave undesirable residues or taints in the end products from the treatment. Also, the choice of solvent mixture may be governed by the desire to leave in the solid product of the treatment such minor components of the raw material such as are related to flavour (taste and smell).

In order that the solvents may penetrate sufficiently into the protein-containing material to remove the fat and the water (and, if desired, the flavour components), it is necessary that the particle size should be suitably small. A particle size of about 0.25 mm. is generally suitable.

In some cases it is difficult, because of the fatty or greasy nature of the protein-containing starting material, to comminute it suitably small. If this is so, then it is sometimes desirable to comminute the starting material to a coarser size for feeding into the counter-current treatment. Then, during the treatment, such as in the third or fourth stage, the material is further comminuted in the presence of the solvents to a smaller size such as 0.25 mm. The finer comminution may be carried out such as by subjecting the mixture of material and solvents to the action of rotating, high-speed cutter blades.

EXAMPLE

Following is one example of a solvent mixture and its operation in this invention:

The solvent consisted of the following mixture:—

- (a) 75 parts by volume of a hydrocarbon solvent of nominal distillation range 54°C—70°C, Specific gravity approximately 0.67, Aromatic content approximately 1%.
- (b) 15 parts by volume ethyl acetate.
- (c) 5 parts by volume acetone.
- (d) 5 parts by volume ethyl alcohol.

This solvent was used in a counterflow treatment of minced beef meat of approximate or nominal composition 60% water, 19% fat, 19% proteinaceous solid matter and 2% minor components related to taste and smell.

The counterflow treatment was carried out in a similar manner as described in our British Patent Specification No. 905,607 in six stages, using 50 gms of minced meat for

each batch of forward flow material and approximately 75 mls of solvent backflow in relation to 50 grams forward flow of material. At each stage the material was agitated in a vessel for approximately five minutes at a temperature approximately 40°C and then drained and squeezed, the solid material being passed to the subsequent stage vessel and the solvent being passed to the previous stage vessel. In the fourth stage the mixture of solid matter and liquor was transferred for comminution to a disintegrator with high speed rotating cutter blades and then transferred back to the fourth stage vessel.

The solid end-product from the sixth or last stage was desolventized by evaporation of the solvent in an oven and then by adding and macerating into it approximately 15% by weight of water and then re-drying. (Note: This procedure is similar to commercial desolventization in which the solvent would be evaporated and recovered by condensation and a small amount of steam would then be used to drive out the last of the solvent vapour from the voids of the solid material). This solid end-product amounted to approximately 9.5 grams from each batch of 50 grams of raw material and it had substantially the normal flavour of the meat. By analysis, the fat content was found to be 1.1%.

The outgoing liquid from the first stage of the treatment consisted of two phases. The one phase was a mixture of water and water-miscible solvent and the other phase consisted of fat dissolved in non-water miscible solvent.

WHAT WE CLAIM IS:—

(1) A process for the production of a substantially de-fatted and de-watered proteinaceous end-product from a protein-containing material of animal, vegetable or marine origin and which contains fat and water, in which process is used a mixture of organic solvents containing one or more solvents substantially miscible with water and one or more solvents substantially non-miscible with water but capable of dissolving fat, the mixtures of solvents having a boiling point or boiling range which lies below the boiling point of water, the protein-containing material being treated by the solvent mixture in a preferably continuous multi-stage counter-current process, of which the first stage is the one where the protein-containing material enters and from which the solvent mixture passes out and the last stage is the one where the new or clean solvent mixture enters and the proteinaceous end-product passes out and that the solvent mixture entering the last stage and passing towards the first stage progressively removes fat and water from the protein-containing

trate moist materials (such as acetone and ethyl alcohol) together with another desirable solvent, such as ethyl acetate. The choice of solvent mixture is governed by, firstly, that it must achieve the essential object of penetrating into the most material to dissolve the fat (or oil) and to liberate the water in a separate phase and, then, by other considerations such as to be recoverable by normal distillation practices for re-use and not to leave undesirable residues or taints in the end products from the treatment. Also, the choice of solvent mixture may be governed by the desire to leave in the solid product of the treatment such minor components of the raw material such as are related to flavour (taste and smell).

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In some cases it is difficult, because of the fatty or greasy nature of the protein-containing starting material, to comminute it suitably small. If this is so, then it is sometimes desirable to comminute the starting material to a coarser size for feeding into the counter-current treatment. Then, during the treatment, such as in the third or fourth stage, the material is further comminuted in the presence of the solvents to a smaller size such as 0.25 mm. The finer comminution may be carried out such as by subjecting the mixture of material and solvents to the action of rotating, high-speed cutter blades.

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WHAT WE CLAIM IS:—

(1) A process for the production of a substantially de-fatted and de-watered proteinaceous end-product from a protein-containing material of animal, vegetable or marine origin and which contains fat and water, in which process is used a mixture of organic solvents containing one or more solvents substantially miscible with water and one or more solvents substantially non-miscible with water but capable of dissolving fat, the mixtures of solvents having a boiling point or boiling range which lies below the boiling point of water, the protein-containing material being treated by the solvent mixture in a preferably continuous multi-stage counter-current process, of which the first stage is the one where the protein-containing material enters and from which the solvent mixture passes out and the last stage is the one where the new or clean solvent mixture enters and the proteinaceous end-product passes out and that the solvent mixture entering the last stage and passing towards the first stage progressively removes fat and water from the protein-containing



- material as it passes from the first stage to the last stage, characterised in that the combined liquid in the first stage exists in two separate phases of which the one phase consists substantially of water-miscible solvent or solvents and water and the other phase consists substantially of non-water-miscible solvent or solvents with fat dissolved therein.
- (2) A process as claimed in Claim 1, further characterised in that, if the protein-containing material before treatment is relatively dry, water is added, preferably to the material before treatment or to the mixture of material and solvents at or near the first stage of treatment.
- (3) A process as claimed in Claim 1 or 2, further characterised in that the solvent mixture is such as also removes taste and smell components from the protein-containing material and which pass out of counter-current apparatus in the combined liquid from the first stage and being all or substantially dissolved in the phase which consists substantially of water-miscible solvent or solvents and water.
- (4) A process as claimed in Claim 1 or 2, wherein the solvent mixture comprises by volume 75 parts hydrocarbon solvent of nominal distillation range 54°C. to 70°C., 15 parts ethyl acetate, 5 parts acetone and 5 parts ethyl alcohol.
- (5) A process as in any one of the preceding claims characterised in that the protein-containing material is subjected to comminution in the presence of the solvents during the counterflow treatment.
- (6) A process as claimed in claim 1 for the production of a substantially de-fatted and de-watered proteinaceous end product from a protein-containing material of animal, vegetable or marine origin substantially as herein described.
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