

SUPERINTENDENCIA DE INDUSTRIA Y COM
División de Nuevas Creaciones
E. S. D.

SUPERINDUSTRIA Y COMERCIO
Radicación : 04109249 00000001 Folios: 56
Fecha (MD): 2005-01-14 16:52:18
Trámite : 002 PATENTES D I REGISTRAR J29 COMPLETEN
Dependencia: 2020 DIVISION DE NUEVAS CREACIONES

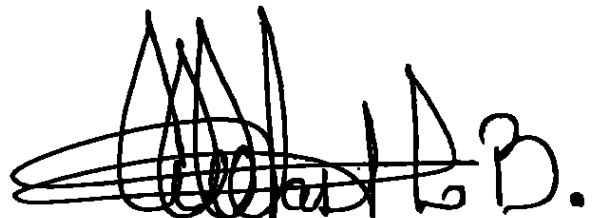
Ref: Expediente No. 04-109249
Solicitud de patente a nombre
de KRAFT FOODS HOLDINGS, INC

En el asunto de la referencia me permito presentar los siguientes documentos:

- 1- La copia certificada de la solicitud Estadounidense No. 10/697.402 del 29 de Octubre de 2003, de la cual se reclamó prioridad. Observo que las reivindicaciones de este documento coinciden con la presente solicitud.
- 2- Cuatro documentos de cesión firmados por cada uno de los inventores legalizados por Apostilla.

Como el expediente queda así completo, pido se ordene la publicación del extracto.

Atentamente,



JERONIMO CASTILLO BONILLA
C.C. 79.462.057 Bogotá
T.P. 104167 C.S. Judicatura

LAW OFFICES

PEDRO CASTILLO PINEDA & ASSOCIATES

P O BOX 48362

Bogotá, Colombia / South America

Power with Assignment

Insert here
Full name
And address
Assignor

The undersigned: AHMAD AKASHE residing at 817 Castleton Court Mundelein Illinois 60060 USA CYNTHIA LYNN JACKSON residing at 2455 Prairie Avenue #3A, Evanston, Illinois 60201 USA; ARIEL S CUDIA residing at 3333 North Hamilton Avenue Chicago Illinois 60618 USA; JOHN WISLER residing at 4730 Crested Butte Trail Rockford Illinois 61114 USA

hereby appoints Jorqueso Castillo Bonilla and/or Ximena Bonilla Pereira and/or Alvaro Castillo Grau domiciled at Calle 93A N° 14-17 of Bogotá, Colombia, jointly, alternatively or indistinctly as attorneys with special and sufficient power to apply and obtain from the competent offices and authorities of Colombia Patent of Invention, Utility Model or Industrial Design for

METHOD OF DEFLAVORING LONEY PROTEIN

Name and
Address of
Assignee

to be issued in the name of KRAFT FOODS HOLDINGS INC Three Lakes Drive Northfield Illinois 60093 USA

to whom assignment has been made of all right and title of said
for the territory of Colombia without any restriction.

The assignor also signs these presents in token of acceptance of the said assignment and hereby authorizes the said attorneys to take all necessary steps before the said offices and authorities for the purpose above stated; to make petitions, declarations and claims and to use all means which said attorneys shall deem proper for the safeguard of the interests of the constituent. This power includes the faculties to receive, desist, renounce, compromise, frigate, substitute it in all or in part, to revoke substitutions, to receive ratifications and to appoint extra-judicial or judicial attorneys. The attorneys mentioned above can exercise this power alternatively or indistinctly

Signatures

AH AKASHE
AHMAD AKASHE

CYNTHIA LYNN JACKSON

ARIEL S CUDIA

JOHN WISLER



SPRINGFIELD, ILLINOIS

APOSTILLE
APOSTILLE

(Convention de La Haye du 5 Octobre 1961)

1. Country: United States of America

This public document

2. has been signed by SALLY A RAKESTRAW

3. acting in the capacity of NOTARY PUBLIC, COOK COUNTY

4. bears the seal/stamp of STATE OF ILLINOIS

Certified

5. Chicago, Illinois

6. DECEMBER 16, 2004

7. by the Secretary of State, State of Illinois

8. No.C04JW27753

9. Seal/Stamp:

10. Signature:

Jesse White

JESSE WHITE
SECRETARY OF STATE
STATE OF ILLINOIS

State of Illinois) ss
County of Cook .)



I hereby certify that before me in the County of Cook, in the State of Illinois, personally appeared AHMAD AKASHE, personally known by me, who then and there was duly sworn by me, and under oath acknowledged that the foregoing Assignment was duly signed, sealed and delivered by him on the date appearing at the foot thereof.

Nov. 17, 2004 Sally A. Rakostian
Notary Public

My Commission Expires 5/2/06

State of Illinois) ss
County of Cook)

I hereby certify that before me in the County of COOK, in the State of Illinois, personally appeared CYNTHIA LYNN JACKSON, personally known by me, who then and there was duly sworn by me, and under oath acknowledged that the foregoing Assignment was duly signed, sealed and delivered by him on the date appearing at the foot thereof.

_____, 2004 _____
Notary Public
My Commission Expires _____

57
5X

LAW OFFICES

PEDRO CASTILLO PINEDA & ASSOCIATES

P.O. BOX 42362

Bogotá, Colombia / South America

Power with Assignment

Insert here
Full name
And address
Assignor

The undersigned: AHMAD AKASHE residing at 817 Castleton Court, Mundelein, Illinois 60060, USA; CYNTHIA LYNN JACKSON residing at 2455 Prairie Avenue, #3A, Evanston, Illinois 60201, USA; ARIEL S. CUDIA residing at 3333 North Hamilton Avenue, Chicago, Illinois 60618, USA; JOHN WISLER residing at 4730 Crested Butte Trail, Rockford, Illinois 61114, USA

hereby appoints Jeronimo Castillo Bonilla and/or Ximena Bonilla Pereira and/or Alvaro Castillo Giraldo domiciled at Calle 93A N° 14-17 of Bogotá, Colombia, collectively, alternatively, or indistinctly as attorneys with special and sufficient powers to apply and obtain from the competent offices and authorities of Colombia Patent of Invention, Utility Model or Industrial Design for

METHOD OF DEFLAVORING WHEY PROTEIN

Name and
Address of
Assignee

to be issued in the name of KRAFT FOODS HOLDINGS, INC., Three Lakes Drive, Northfield, Illinois 60093 USA

to whom assignment has been made of all right and title of said for the territory of Colombia without any restriction.

The assignee also signs these presents in token of acceptance of the said assignment and hereby authorizes the said attorneys to take all necessary steps before the said offices and authorities for the purpose above stated; to make petitions, descriptions and claims and to use all means which said attorneys shall deem proper for the safeguard of the interests of the assignee. This power includes the faculties to receive, desert, renounce, compromise, transact, substitute it in all or in part, to revoke substitutions, to receive notifications and to appoint extra-judicial, or judicial attorneys. The attorneys mentioned above can exercise this power alternatively or indistinctly.

AHMAD AKASHE


CYNTHIA LYNN JACKSON 11/17/04

ARIEL S. CUDIA

JOHN WISLER



SPRINGFIELD, ILLINOIS

APOSTILLE
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(Convention de La Haye du 5 Octobre 1961)

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Certified

5. Chicago, Illinois

6. DECEMBER 16, 2004

7. by the Secretary of State, State of Illinois

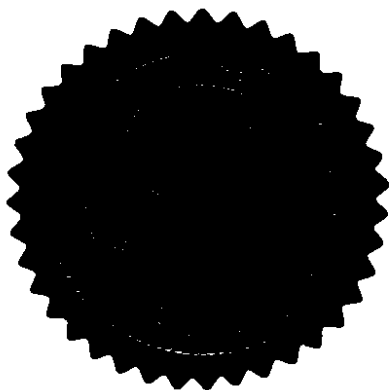
8. No.C04JW27751

9. Seal/Stamp:

10. Signature:

Jesse White

JESSE WHITE
SECRETARY OF STATE
STATE OF ILLINOIS



54

State of Illinois)

County of Cook)

ss

I hereby certify that before me in the County of Cook, in the State of Illinois, personally appeared AHMAD AKASHE, personally known by me, who then and there was duly sworn by me, and under oath acknowledged that the foregoing Assignment was duly signed, sealed and delivered by him on the date appearing at the foot thereof.

_____, 2004

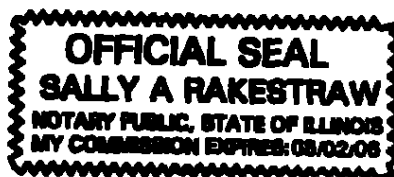
Notary Public

My Commission Expires _____

State of Illinois)

County of Cook)

ss



I hereby certify that before me in the County of COOK, in the State of Illinois, personally appeared CYNTHIA LYNN JACKSON, personally known by me, who then and there was duly sworn by me, and under oath acknowledged that the foregoing Assignment was duly signed, sealed and delivered by him on the date appearing at the foot thereof.

Nov. 17

, 2004

Sally A. Rakestraw
Notary Public

My Commission Expires

5/2/06

LAW OFFICES

PEDRO CASTILLO PINEDA & ASSOCIATES

P.O. BOX 48362

Bogotá, Colombia / South America

Power with Assignment

Insert here
Full name
And address
Assignor

The undersigned: AHMAD AKASHE residing at 817 Castleton Court, Mundelein, Illinois 60060, USA; CYNTHIA LYNN JACKSON residing at 2455 Prairie Avenue, #3A, Evanston, Illinois 60201, USA; ARIEL S. CUDIA residing at 3333 North Hamilton Avenue, Chicago, Illinois 60618, USA; JOHN WISLER residing at 4730 Crested Butte Trail, Rockford, Illinois 61114, USA;

hereby appoints ~~Jesús~~ Castillo Bonilla and/or Ximena Bonilla Perdomo and/or Alvaro Castillo Gaitanopoulos at Calle 93A N° 14-17 of Bogotá, Colombia, jointly, alternatively or indistinctly as attorneys with special and sufficient power to apply and obtain from the competent offices and authorities of Colombia Patent of Invention, Utility Model or Industrial Design for

METHOD OF DEFLAVORING WHEAT PROTEIN

Name and
Address of
Assignee

to be issued in the name of KRAFT FOODS HOLDINGS, INC., Three Lakes Drive, Northfield, Illinois 60093 USA

to whom assignment has been made of all right and title of said
for the territory of Colombia without any restriction.

The assignee also signs these presents in token of acceptance of the said assignment and hereby authorizes the said attorneys to take all necessary steps before the said official and authorities for the purpose above stated; to make petitions, declarations and claims and to use all means which said attorneys shall deem proper for the safeguard of the interests of the constituent. This power includes the faculties to receive, desert, renounce, compromise, transmit, substitute in all or in part, to revoke substitutions, to receive notifications and to appoint extra-judicial or judicial attorneys. The attorneys mentioned above can exercise this power alternatively or indistinctly.

Signatures

✓

AHMAD AKASHE

✓

CYNTHIA LYNN JACKSON

✓

ARIEL S. CUDIA

✓

JOHN WISLER



SPRINGFIELD, ILLINOIS

APOSTILLE

APOSTILLE

(Convention de La Haye du 5 Octobre 1961)

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3. acting in the capacity of NOTARY PUBLIC, COOK COUNTY

4. bears the seal/stamp of STATE OF ILLINOIS

Certified

5. Chicago, Illinois

6. DECEMBER 16, 2004

7. by the Secretary of State, State of Illinois

8. No.C04JW27750

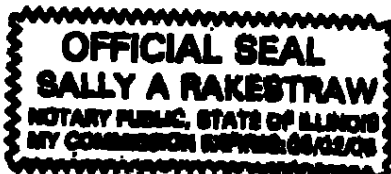
9. Seal/Stamp:

10. Signature:

Jesse White

JESSE WHITE
SECRETARY OF STATE
STATE OF ILLINOIS

State of Illinois)
County of Cook) ss



I hereby certify that before me in the County of COOK, in the State of Illinois, personally appeared ARIEL S. CUDIA, personally known by me, who then and there was duly sworn by me, and under oath acknowledged that the foregoing Assignment was duly signed, sealed and delivered by him on the date appearing at the foot thereof.

Nov. 18, 2004 Sally A. Rakestraw
Notary Public
My Commission Expires 5/2/06

State of Illinois)
County of Cook) ss

I hereby certify that before me in the County of COOK, in the State of Illinois, personally appeared JOHN WISLER, personally known by me, who then and there was duly sworn by me, and under oath acknowledged that the foregoing Assignment was duly signed, sealed and delivered by him on the date appearing at the foot thereof.

_____, 2004 _____
Notary Public
My Commission Expires _____

LAW OFFICES
PEDRO CASTILLO PINEDA & ASSOCIATES
P O BOX 48362
Bogotá, Colombia / South America

Power with Assignment

Insert here
Full name
And address
Assignor

The undersigned: AHMAD AKASHE residing at 817 Castleton Court Mundelein Illinois 60060 USA, CYNTHIA LYNN JACKSON residing at 2455 Prairie Avenue, #3A, Evanston, Illinois 60201 USA; ARIEL S CUDIA residing at 3333 North Hamilton Avenue Chicago Illinois 60618 USA
JOHN WISLER residing at 4730 Crested Butte Trail Rockford Illinois 61114 USA

hereby appoints ~~Jacques~~ Castillo Bonilla and/or ~~Ximena~~ Bonilla Parola and/or ~~Alvaro~~ Castillo ~~Garcia~~ domiciled at Calle 93A N° 14-17 of Bogotá, Colombia, ~~conjunctly, alternatively or indistinctly~~ as attorneys with special and sufficient power to apply and obtain from the competent offices and authorities of Colombia Patent of Invention, Utility Model or Industrial Design for

METHOD OF DEFLAVORING WHEY PROTEIN

Name and
Address of
Assignee

to be issued in the name of KRAFT FOODS HOLDINGS INC Three Lakes Drive Northfield Illinois 60093 USA

to whom assignment has been made of all right and title of said
for the territory of Colombia without any restriction.

The assignor ~~also~~ ~~signs~~ ~~these~~ ~~presently~~ in token of acceptance of the said assignment and ~~highly~~ ~~authorizes~~ the said attorneys to take all necessary steps before the said offices and authorities for the purpose above stated; to make petitions, descriptions and claims and to use all means which said attorneys shall deem proper for the safeguard of the interests of the continent. This power includes the faculties to receive, decline, renounce, compromise, transmit, substitute it in all or in part, to revoke substitutions, to receive notifications and to appoint extra-judicial or judicial attorneys. The attorneys mentioned above can exercise this power alternatively or indistinctly

Signatures

✓

AHMAD AKASHE

✓

CYNTHIA LYNN JACKSON

✓

ARIEL S CUDIA

✓ *John Wisler*
JOHN WISLER



SPRINGFIELD, ILLINOIS

APOSTILLE

APOSTILLE

(Convention de La Haye du 5 Octobre 1961)

1. Country: United States of America

This public document

2. has been signed by SALLY A RAKESTRAW

3. acting in the capacity of NOTARY PUBLIC, COOK COUNTY

4. bears the seal/stamp of STATE OF ILLINOIS

Certified

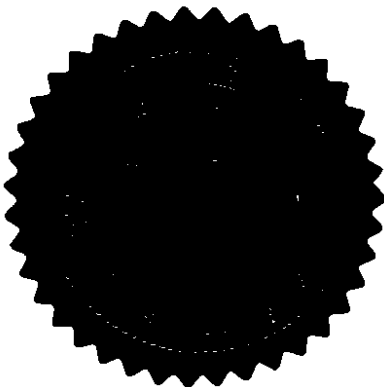
5. Chicago, Illinois

6. DECEMBER 16, 2004

7. by the Secretary of State, State of Illinois

8. No.C04JW27752

9. Seal/Stamp:



10. Signature:

Jesse White

JESSE WHITE
SECRETARY OF STATE
STATE OF ILLINOIS

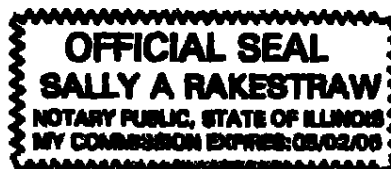
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State of Illinois)) ss
County of Cook)

I hereby certify that before me in the County of COOK, in the State of Illinois, personally appeared ARIEL S. CUDIA, personally known by me, who then and there was duly sworn by me, and under oath acknowledged that the foregoing Assignment was duly signed, sealed and delivered by him on the date appearing at the foot thereof.

_____, 2004 _____
Notary Public
My Commission Expires _____

State of Illinois)) ss
County of Cook)



I hereby certify that before me in the County of COOK, in the State of Illinois, personally appeared JOHN WISLER, personally known by me, who then and there was duly sworn by me, and under oath acknowledged that the foregoing Assignment was duly signed, sealed and delivered by him on the date appearing at the foot thereof.

Rec 1, 2004 Sally A. Rakestraw
Notary Public
My Commission Expires 5/2/06



THE UNITED STATES OF AMERICA

TO ALL TO WHOM THESE PRESENTS SHALL COME:

UNITED STATES DEPARTMENT OF COMMERCE

United States Patent and Trademark Office

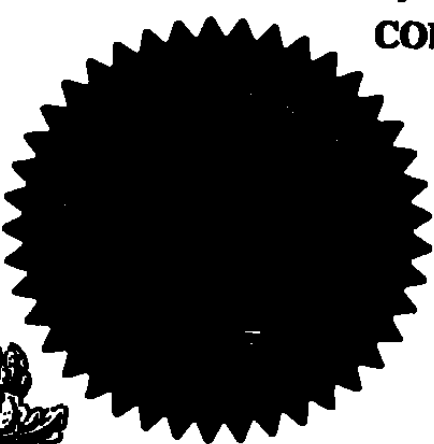
November 18, 2004

THIS IS TO CERTIFY THAT ANNEXED HERETO IS A TRUE COPY FROM THE RECORDS OF THE UNITED STATES PATENT AND TRADEMARK OFFICE OF THOSE PAPERS OF THE BELOW IDENTIFIED PATENT APPLICATION THAT MET THE REQUIREMENTS TO BE GRANTED A FILING DATE UNDER 35 USC 111.

APPLICATION NUMBER: 10/697,402

FILING DATE: October 29, 2003

**By Authority of the
COMMISSIONER OF PATENTS AND TRADEMARKS**




M. K. HAWKINS
Certifying Officer

13049 U.S. PTO



UTILITY PATENT APPLICATION TRANSMITTAL
(for Noncontinuing, Nonprovisional Applications
under 37 C.F.R. §1.53(b))

Attorney Docket No. 77016

Customer No. 22242

22141 U.S. PTO
10/697402



Mail Stop PATENT APPLICATION
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

Transmitted herewith for filing under 37 C.F.R.
§1.53(b) is the nonprovisional, noncontinuing
patent application for:

Title: METHOD OF DEFLAVORING WHEY
PROTEIN

First Named Inventor or Application Identifier:
AKASHE et al.

CERTIFICATE OF MAILING BY "EXPRESS MAIL"

"Express Mail" Mailing Label Number

EV 323484245 US

Date of Deposit October 29, 2003

I hereby certify that this paper or fee is being deposited
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Edward Price

(Typed or printed name of person mailing)

Edward Price
(Signature of person mailing)

- ☒ 31 pages of the specification (including claims) are enclosed.
- ☒ 2 sheet(s) of drawings are enclosed. ☐ Formal ☒ Informal
- ☐ An executed Oath or Declaration and Power of Attorney naming the actual inventors is enclosed.
- ☒ The names of persons believed to be the actual inventors are set forth in the enclosed unexecuted Oath or Declaration and Power of Attorney (§1.41(a) and §1.53(b)).
- ☐ An Assignment of the invention to _____, and cover sheet are enclosed.
- ☐ A check in the amount of \$_____ to cover the fee for recording the assignment is enclosed.
- ☐ The Recordation Form Cover Sheet includes authorization to charge our Deposit Account for recording the assignment.
- ☐ A 37 C.F.R. §3.73(b) statement is enclosed where an assignee seeks to take action.

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Attorney Docket No. 77018

- ☒ An Information Disclosure Statement is enclosed.
- ☐ A Form PTO/SB/08 is enclosed.
- ☐ _____ References (copies) listed on the Form PTO/SB/08 are enclosed.
- ☒ A Return Receipt Postcard is enclosed (MPEP §503).
- ☐ Priority of application number _____ filed on _____ in _____ is claimed under 35 U.S.C. §119.
- ☐ A certified copy of the priority document is enclosed.
- ☐ A Computer Program Listing Appendix is enclosed.
- ☐ A Transmittal Cover Letter for Computer Program Listing Appendix is enclosed.
- ☐ Two (2) Compact Discs are enclosed.
- ☐ A Nucleotide and/or Amino Acid Sequence Submission is enclosed.
- ☐ A Computer Readable Copy is enclosed.
- ☐ A Paper Copy (Identical to Computer Copy) is enclosed.
- ☐ A Statement Verifying Identity of above Copies is enclosed.
- ☒ The filing fee is calculated below:

Fee Calculation For Claims As Filed

Basic Fee							\$ 770.00
Independent Claims	2	-	3	=	0	x \$ 86.00	= \$ 0.00
Total Claims	32	-	20	=	12	x \$ 18.00	= \$ 216.00
Fee for Multiply Dependent Claims						\$ 290.00	
Total Filing Fee							\$ 986.00

- ☐ Applicant(s) assert entitlement to Small Entity Status (37 C.F.R. § 1.27), reducing the Filing Fee by half to: \$ 0.00
- ☐ A check in the amount of \$_____ to cover the filing fee is enclosed.
- ☒ Charge \$ 986.00 to Deposit Account No. 06-1135.
- ☐ The payment of the Filing Fee is to be deferred until the Declaration is filed. Do not charge our Deposit Account.

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Attorney Docket No. 77016

☒ A separate written request under 37 C.F.R. §1.136(a)(3), which is a general authorization to treat any concurrent or future reply requiring a petition for an extension of time under 37 C.F.R. §1.136(a) for its timely submission as incorporating a petition for an extension of time for the appropriate length of time, is enclosed.

☒ The Director is hereby authorized to charge any additional fees which may be required in this application under 37 C.F.R. §§1.16-1.17 during its entire pendency, or credit any overpayment, to Deposit Account No. 06-1135. Should no proper payment be enclosed herewith, the Director is authorized to charge the unpaid amount to Deposit Account No. 06-1135. This sheet is filed in triplicate.

☐ Also enclosed:

☒ Address all future communications to CUSTOMER NUMBER 22242.

October 29, 2003

Date



Richard A. Kaba

Registration No. 30,562

FITCH, EVEN, TABIN & FLANNERY
120 South LaSalle Street, Suite 1600
Chicago, Illinois 60603-3406
Telephone: (312) 577-7000
Facsimile: (312) 577-7007

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METHOD OF DEFLAVORING WHEY PROTEIN

The present application is a continuation-in-part application of United States Patent Application Serial Number 09/939,500, filed August 23, 2001, which was based on, and claimed benefit of, United States Provisional Application Serial Number 60/250,228, filed on November 30, 2000, both of which are hereby incorporated by reference.

BACKGROUND OF THE INVENTION

This invention relates generally to the processing of whey protein-containing materials for use in various food products. More particularly, the invention relates to a method of deflavoring whey protein materials in order to make them acceptable in a wide range of foods.

In recent years, whey proteins have become widely used in food products, for the health benefits to be obtained from their use. For example, studies suggest that whey proteins may provide a variety of health benefits such as anti-hypertension activity, immune system enhancement, antimicrobial activity, intestinal health enhancement, and oral hygiene activity. In some applications, the taste of the whey protein materials is not objectionable. However, in some uses, such as dairy analog products, beverages and the like, and especially when the amount of whey protein is increased to the levels associated with such health benefits, the flavors found in whey protein materials may prevent their ready acceptance by the consumer. Thus, in order to extend the uses of whey protein materials, the present inventors wanted to find a method of reducing the flavor components of whey protein materials. However, it was not evident that methods which had been used previously to remove flavor components from other organic materials would be successful in the treating of whey protein materials. Organic materials, since they have complex compositions, must be tested to determine whether any given method of treating them will be satisfactory.

One example of previously employed methods to purify organic materials is found in U.S. Patent 4,477,480, in which the patentees show that

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starch can be treated with an alkali to remove objectionable flavor components. In a commonly assigned patent, U.S. Patent 4,761,186, ultrafiltration is used to purify starch. In both cases, flavor components are removed from the starch, in the '480 patent by solubilizing the flavor components so that they can be washed out of the relatively insoluble starch. In the '186 patent, ultrafiltration was used to remove the flavor components as permeate, while the insoluble starch remained in an aqueous slurry. By contrast, the present invention separates flavor components from soluble high molecular weight whey proteins.

There are many articles and patents which relate to processing soy materials in order to recover the protein content and which at the same time reduce the flavor compounds to make the proteins more acceptable in food products. However, these previous disclosures were not specifically directed to removal of flavoring compounds and recovering as much of the protein as possible. One example is U.S. Patent 4,420,425 in which protein components of soy are solubilized at a pH of 7 to 11, preferably about 8 and, after ultrafiltration through a membrane having a molecular weight cut off above 70,000, are recovered by spray drying the retained soy proteins. In variants, only a portion of the protein is solubilized at lower pH values and subjected to ultrafiltration with a membrane having a cutoff preferably above 100,000 molecular weight, the product was found to have improved color and flavor. A higher cutoff valve would be expected to result in a loss of valuable proteins. In another patent, U.S. Patent 5,658,714, a soy flour slurry is pH-adjusted to the range of 7 to 10 to solubilize proteins, which are then passed through an ultrafiltration membrane and phytate and aluminum are retained, presumably as solids. While the molecular weight cutoff of the membrane was not given, it is assumed that the pore size was large in order to be able to pass the soluble proteins. Both of these patents contain extensive discussions of the efforts of others in the processing of soy materials; neither teaches or suggests the control of pH during the ultrafiltration process.

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In a group of related patents, Mead Johnson Company disclosed processes for solubilizing soy proteins by raising the pH of an aqueous solution of soy materials and recovering the proteins which are said to have a bland taste. The processes are principally directed to concentrating proteins rather than removing flavor compounds. In U.S. Patent 3,995,071, the pH was increased to 10.1 to 14 (preferably 11 to 12) to solubilize soy proteins, after which the pH was lowered to about 6 to 10 and ultrafiltration with a membrane having a molecular weight cutoff of 10,000 to 50,000 Daltons was used to retain the proteins while discarding carbohydrates and minerals. In U.S. Patent 4,072,670, emphasis was placed on removing phytates and phytic acid by solubilizing proteins at a pH of 10.6 to 14 and a temperature of 10 to 50°C to make the phytates and phytic acid insoluble, then separating them and finally acidifying the solution to a pH of about 4 to 5 to precipitate the soy proteins. In U.S. Patent 4,091,120 soy proteins were solubilized at a pH less than 10, preferably 7 to 9, and ultrafiltration was used to separate the proteins as retentate, while passing carbohydrates as permeate. These patents do not teach or suggest control of the pH during the ultrafiltration process.

The present inventors wanted to remove compounds in soy protein materials which contribute color and flavor and which interfere with the use of soy protein in certain food products such as beverages, dairy analogs, and the like. They found that soy protein-derived materials can be treated successfully, recovering substantially all of the proteins and rejecting the compounds which cause undesirable color and flavor. Moreover, by controlling the pH within the range of about 8.5 to about 12 during the ultrafiltration process, deflavored soy protein materials having improved functional properties can be obtained. Thus, the product is suitable for many food products. Now the present inventors have surprisingly discovered that a related process can be advantageously applied to whey protein materials to remove undesirable flavor components to obtain a deflavored whey protein material which can be incorporated into many different food products. The

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process can, however, be modified such that it can be operated in a basic or acid pH range. Thus, either basic or acidic deflavored soy protein can be prepared using the process of this invention.

SUMMARY OF THE INVENTION

5 Broadly, the invention is a process for preparing an aqueous whey composition having a whey protein concentration of about 1 to about 50 percent, which is pH-adjusted to solubilize the whey protein content and to release the flavoring compounds. Then the composition is subjected to ultrafiltration, while maintaining pH control, using a membrane capable of
10 retaining substantially all of the protein content of the whey protein material while removing flavoring components as permeate. As noted above, the present inventors have now surprisingly discovered that a method used for deflavoring soy protein materials can be used in a similar manner for deflavoring whey protein materials. The present process, as applied to whey
15 proteins, can be run under either acidic or basic conditions as desired to produce either an acidic or a basic deflavored whey protein material.

 The deflavored whey protein materials prepared by the present methods are ideally suited for use in dairy and non-dairy beverages, smoothies, health drinks, confectionary type products, nutritional bars,
20 cheeses, cheese analogs, dairy and non-dairy yogurts, meat and meat analog products, cereals, baked products, snacks, and the like. Preferably the acidic deflavored whey protein is used in acidic food products and the basic deflavored whey protein is used in neutral and basic food products. Thus, by proper selection, one can avoid destabilizing the deflavored whey protein
25 associated with passing it through its isoelectric point.

 The present invention provides methods for deflavoring whey proteins. In addition to the removal of off-flavors, the present invention also allows the efficient removal of lactose, thereby allowing concentration of the whey proteins to high levels. Typically whey protein contains about 70 to about 80
30 percent (dry basis) lactose. Generally, levels of lactose less than about 15

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percent (dry basis) can be obtained in the deflavored whey protein. By
extensive ultrafiltration/dilfiltration (i.e., greater than 5 wash cycles and
typically in the range of about 6 to 7 wash cycles), the level of lactose can be
reduce to less than about 99 percent (dry basis). Moreover, the deflavored
5 whey protein materials can be prepared containing greater than about 50
percent protein (on a dry basis), and preferably about 65 to about 95 percent
protein; obtaining higher levels of protein (generally greater than about 85
percent) require extensive ultrafiltration/dilfiltration. Thus, it becomes
possible to incorporate whey protein in conventional food products at
10 sufficiently high levels (generally sufficient to provide about 2.5 to about 20 g
whey protein per single serving size (generally about 25 to about 100 g for
solid food products and about 100 to about 300 ml for liquid food products))
to provide health benefits of whey protein. Prior to this invention,
Incorporation whey proteins at such levels generally resulted in significant off-
15 flavors and, therefore, only very limited acceptance by consumers. Moreover,
the removal of lactose may allow the use of such food products by lactose-
intolerant consumers; generally, in such cases, at least about 95 percent of
the lactose should be removed.

In one aspect, the invention is a method of deflavoring whey protein
20 materials, which method includes preparing an aqueous composition of the
whey protein material containing flavoring compounds, adjusting the pH to a
basic range of about 8.5 to about 12 or to an acidic range of about 2.4 to
about 4 to solubilize the protein content of the whey protein material and
release the flavor components, and then passing the pH-adjusted
25 composition adjacent to an ultrafiltration membrane having pores which
provide a molecular weight cutoff up to 50,000 Daltons, while maintaining the
pH in the same range as to which the aqueous composition was adjusted,
thus retaining substantially all of the whey protein content, while passing
through the pores the flavor producing compounds.

30 In another aspect (herein termed the "basic mode of operation"), the
invention includes adjusting the pH to the range of about 8.5 to about 12 with

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an alkali such as sodium, potassium or calcium hydroxides to maintain the solubility of the whey protein content and release the flavor compounds, making it possible to separate such compounds by ultrafiltration. Importantly, the pH in this basic mode of operation is also controlled within the range of about 8 to about 12 during the ultrafiltration process.

In another aspect (herein termed the "acidic mode of operation"), the invention includes adjusting the pH to the range of about 2.5 to about 4 with an edible acid (e.g., citric acid, acetic acid, lactic acid, malic acid, ascorbic acid, fumaric acid, adipic acid, phosphoric acid, sodium hydrogen sulfate, and the like) to maintain the solubility of the whey protein content and release the flavor compounds, making it possible to separate such compounds by ultrafiltration. The preferred edible acids for use in this acidic mode of operation include phosphoric acid, citric acid, and malic acid. Importantly, the pH in this acidic mode of operation is also controlled within the range of about 2.5 to about 4 during the ultrafiltration process.

Native whey proteins (i.e., undenatured) are generally soluble over a wide range of pH values. Denaturing of such protein, as often occurs during processing (e.g., cheese manufacture, pasteurization, elevated temperature, ultrafiltration, and the like) have decrease solubility (especially around the isoelectric point of about 7.4). Maintaining the pH of the deflavored whey protein in essentially the same range as its ultimate use in a food product allows the maintenance of desired solubility. Using a deflavored whey protein prepared using the basic mode of operation in a neutral or basic food product and using a deflavored whey protein prepared using the acidic mode of operation in an acidic food products avoids modifying the pH of the deflavored whey protein (and passing it through its isoelectric point) and thereby provides maximum solubility in the food product.

In one embodiment, the invention is a method for deflavoring whey protein materials in a continuous process wherein a pH-adjusted aqueous mixture of whey protein materials is passed adjacent an ultrafiltration membrane to separate the flavor components. The pH is maintained at about

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8.5 to about 12 for the basic mode of operation or at about 2.5 to about 4 for the acidic mode of operation during the ultrafiltration by the addition of the appropriate amount of an appropriate pH-altering material (i.e., a base or acid depending on the desired mode of operation). The permeate containing
5 flavor components, lactose, minerals, and water is passed adjacent a reverse osmosis membrane to dewater the permeate and the separated water is recycled to join recycled retentate and fresh pH-adjusted whey materials. A portion of the retentate is continually removed and the deflavored whey protein materials recovered.

10 In a preferred embodiment, the invention is a method for deflavoring whey protein materials in a batch or semi-continuous process wherein a pH-adjusted aqueous mixture of whey protein materials is passed adjacent an ultrafiltration membrane, the permeate is separated for recovery of the flavor components, and the retentate is recycled to join fresh pH-adjusted whey
15 protein materials. Water is added periodically or continuously to replace the water lost to the permeate and to adjust the concentration of whey materials in the combined stream to a predetermined level. If necessary, a pH-altering material (e.g., a base or an acid) can be added to the recycled retentate or added water to control the pH to the desired range during the ultrafiltration
20 process. The process is continued until essentially all of the flavoring compounds have been removed. If desired, the process can also be continued until sufficient levels of lactose removal have been obtained; such reduced lactose materials may be used in food products directed to lactose-intolerant individuals.

25 In another preferred embodiment, the present invention provides a method for preparing deflavored whey protein material, said method comprising:

(a) preparing an aqueous composition of a whey protein material containing soluble whey proteins and flavoring compounds;

(b) adjusting the aqueous composition of (a) to either (1) a basic pH in the range of about 8.5 to about 12 or (2) an acidic pH in the range of about 2.5 to about 4;

(c) passing the aqueous composition of (b) adjacent an ultrafiltration membrane having a molecular weight cutoff up to about 50,000 Daltons, while maintaining the pH in the same range as adjusted in step (b), under suitable ultrafiltration conditions wherein the flavor compounds pass through the membrane, thereby deflavoring the whey protein material and retaining substantially all of the soluble whey proteins; and

(d) recovering the soluble whey proteins retained by the ultrafiltration membrane to obtain the deflavored whey protein material. It is generally preferred that any insoluble materials be removed from the whey protein composition prior to the ultrafiltration step. Removal of such insoluble materials (which could include, for example cheese fines, fat globules, and casein aggregates using whey derived from a cheese making process) could be carried out at any time before ultrafiltration step, including before the preparing the initial aqueous solution of step (a).

The ultrafiltration membrane used in the method of the invention will have a molecular weight cutoff up to 50,000 Daltons, preferably 1,000 to 50,000, most preferably about 10,000.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 is a block diagram of one process employing the invention.

FIG. 2 is a block diagram of a preferred embodiment of the invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Whey Protein Materials. Whey proteins have high nutritive value for humans. In fact, the amino acid composition of such whey proteins is close to an ideal composition profile for human nutrition. Unfortunately, use of such whey proteins in food compositions have been limited due to undesirable odors and/or flavors as well as other organoleptic problems associated with whey proteins. Normal whey protein materials generally have significant

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lactose, milky, and animal flavors which can impact the flavor profiles of food products, especially otherwise bland food products. Off flavors in whey protein materials are generally attributed to lipid oxidation of unsaturated fatty acids during, or after, the cheese making process and/or to Maillard browning reactions. Lipid oxidation can result in formation of volatile aldehydes, ketones, esters, and alcohols, which appear to contribute to a cardboard-like flavor. Typically, whey protein materials contribute, or provide, off flavors described as carboardy, musty, metallic, sour, cooked, astringent, and diacetyl; see, for example, Laye et al., *Milchwissenschaft*, 50, 268-272 (1995); Carunchia Whetstine et al., *J. Dairy Sci.*, 86, 439-448 (2003).

The present inventors have found that defects normally associated with whey proteins can be significantly reduced, and in some cases eliminated, using the process of this invention. Deflavored whey protein as produced by the present invention can not only be used in a wide variety of food products, but they can be used in higher levels that has been possible before, thereby providing nutritionally superior food products. Deflavored whey proteins can be prepared from, for example, whey obtained from conventional cheese making processes, whey protein isolate, whey protein concentrate, and the like.

Ultrafiltration Membranes. Filtration is used to separate many materials. In the present invention, ultrafiltration is used to remove flavoring compounds from whey protein materials. Importantly, the pH of the whey protein material should be maintained in the range of about 8 to about 12 during the ultrafiltration process. Ultrafiltration is intended to remove particles having a size between 10 to 1,000 Angstroms (0.001 to 0.1 μm), corresponding generally to particles having a molecular weight between 10,000 and 1,000,000, and which may also be affected by the shape of such high molecular weight particles. Whey proteins have molecular range between about 14,000 and 100,000. A typical analysis of whey proteins is provided in the table below:

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Whey Protein Fraction	MW (daltons)	Percentage of Total Whey Proteins
Beta-lactoglobulins	18300	50
Alpha-lactalbumin	14000	12
Immunoglobulins	15000 - 100,000	10
Bovine Serum Albumin	69000	5
Proteose-peptones	4100 - 41,000	23

A membrane may be chosen which is capable of passing all of the whey proteins or only a selected portion. In the present invention, the whey proteins are retained by the ultrafiltration membrane under the selected operating conditions, while the lower molecular weight flavoring compounds pass through the membrane and are separated, thus improving the color and flavor of the retained whey proteins and associated solids.

A polymer ultrafiltration membrane may be defined as an anisotropic (non-uniform) layer. One face is a skin containing pores which determine the size of molecules which can pass through the membrane. Supporting the surface skin is a spongy structure which extends to the opposite face. Such membranes are commonly made by coagulation of polymers in an aqueous bath. Typical polymers which are used include polysulfones, cellulose esters, poly(vinylidene fluoride), poly (dimethylphenylene oxide), poly (acrylonitrile), which can be cast into membranes. Often, the membranes are formed into hollow tubes which are assembled into bundles, through which the solution to be filtered is passed. Alternatively, flat membrane sheets and spiral designs may be used. In commercial practice, pressure is applied to facilitate movement of the lower molecular weight compounds through the membrane. The membrane must be able to withstand the pressures used, making it important that the spongy supporting structure be uniform to avoid breaking the surface skin and bypassing the membrane.

In addition to the polymeric membranes just described, other materials have been used to make ultrafiltration membranes, such as ceramics, sintered metals, and other inorganic materials. The present invention is not

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limited to any particular type of membrane. In general, the membrane must be able to pass the flavoring compounds, which are believed to have molecular weights lower than 1,000 Dalton. More importantly, the membranes must be able to retain substantially all of the solubilized whey proteins. Thus, the membrane of the invention will have a molecular weight cutoff up to about 50,000 Daltons, preferably about 1,000 to 50,000 Daltons, more preferably 10,000 to 30,000 Daltons.

Process. The process of the invention includes the following steps:

(1) Prepare an aqueous mixture of the whey protein material. In cases where the original whey protein material is an aqueous solution (e.g., whey from a cheese manufacturing process), the material may be used as is, or additional water may be added or removed as desired, to form the aqueous mixture. For dried whey materials (e.g., whey protein isolate, whey protein concentrate, and the like), water will, of course, need to be added to form the aqueous mixture.

(2) Determine whether to employ the basic or acid mode of operation. This determination will normally depend on the anticipated end use of the deflavored whey protein. If the deflavored whey protein is intended to be used in a food product normally having a neutral or basic pH, the basic mode of operation will be preferred. If the deflavored whey protein is intended to be used in a food product normally having an acidic pH, the acidic mode of operation will be preferred. For the basic mode of operation, add a base to raise the pH of the aqueous mixture to about 8.5 to about 12 in order to maintain the solubility of the whey proteins and to release the flavoring compounds. For the acidic mode of operation, add an acid to lower the pH of the aqueous mixture to about 2.5 to about 4 in order to maintain the solubility of the whey proteins and to release the flavoring compounds.

(3) Pass the pH-adjusted mixture, while maintaining the pH in the same range as used in step (2) above, adjacent to an ultrafiltration membrane having a molecular weight cutoff up to about 50,000 Daltons, remove the flavoring compounds as permeate, and remove the remaining whey proteins and other whey materials as retentate.

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(4) Neutralize the retentate and recover the whey proteins.

All types of whey protein materials are considered to be potential sources of whey protein for use in the present invention and ultimately for use in food products. Thus, for example, suitable whey protein materials includes whey obtained from conventional cheese making processes, whey protein isolate, whey protein concentrate, and the like. Thus, whey protein materials which contain whey proteins are provided in or combined into an aqueous mixture, generally a slurry of whey protein solids. The protein content is needed for food products, but as discussed above, it is believed to contain flavoring compounds which must be released in order that they can be separated. The separation of flavoring compounds is carried out in an aqueous mixture in which both the proteins and flavoring compounds are dissolved. The concentration of the whey protein materials in the aqueous mixture will be in the range of about 1 to about 50 percent. Generally, the concentration of whey protein materials after pH adjustment will change during the subsequent ultrafiltration step as water is removed with the permeate. The water will be replaced either periodically or continuously. For example, in diafiltration water is added to gradually dilute the retained proteins in a batch or semi-continuous process.

The second step, as will be seen in the examples, is important if removal of the flavoring compounds is to be accomplished. The whey proteins are solubilized by adding the appropriate pH-modifying agent base to the aqueous mixture to achieve either a pH of about 8.5 to about 12 (the basic mode of operation) or a pH of about 2.5 to about 4 (the acidic mode of operation).

For the basic mode of operation, it has been found that a pH of about 8.5 or above is needed to maintain the solubility of the whey proteins during ultrafiltration, while a pH higher than about 12 is likely to cause undesirable degradation of the proteins. While in theory, any base might be used, sodium or potassium hydroxide are preferred, particularly potassium hydroxide. Other bases which may have application include calcium, magnesium and ammonium hydroxides. For the acidic mode of operation, while in theory any

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edible acid might be used, phosphoric acid, citric acid, and malic acid are preferred, with phosphoric acid being particularly preferred.

It is believed that maintaining the solubility of solubilizing the whey proteins during ultrafiltration allows the flavoring compounds to remain in a form in which they can be removed; if the whey protein denature during ultrafiltration, the flavoring compounds may become bound or encapsulated by the whey proteins, thereby preventing or reducing the effectiveness of their removal. The flavoring compounds, which have relatively low molecular weight compared to the whey proteins are able to pass through the pores of the ultrafiltration membrane, while substantially all of the solubilized whey proteins are too large and are retained. Importantly, the pH should be maintained within the just described ranges (i.e., about 8.5 to about 12 for the basic mode of operation or about 2.5 to about 4 for the acidic mode of operation) during the ultrafiltration/diafiltration process to allow as much of the flavoring compounds as possible to be removed.

The third step could be carried out in a batch manner similar to the laboratory experiments reported below in Examples 1-5 in which the flavor compounds and water passed through the membrane and were removed by flowing water. However, in commercial applications of the process of the invention, the pH-adjusted aqueous mixture would be circulated continuously adjacent to an ultrafiltration membrane. Since water, the pH-modifying agent, and the flavoring compounds pass through the membrane as permeate and are discarded, additional water will be added to maintain the desired concentration of whey protein materials, which will tend to change the pH of the aqueous mixture. This water may be augmented by dewatering the permeate and recycling the recovered water to the feed stream. A pH-modifying material (e.g., base or acid as appropriate) can be added as necessary to control the pH in the desired range (i.e., about 8.5 to about 12 for the basic mode of operation or about 2.5 to about 4 for the acidic mode of operation)) directly to the ultrafiltration solution, to any recycled aqueous material, or to makeup water as desired.

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After removal of the flavoring compounds (i.e., after completion of the ultrafiltration process), further neutralization of the filtered solution may be accomplished by withdrawing product and adding an acid as required to reach the desired pH. After pH adjustment, the aqueous mixture of whey proteins and other materials may be used directly in food products, or it may be concentrated or dried as required for the intended use.

A process for deflavoring whey protein materials by ultrafiltration may be operated in various ways. The pH during the ultrafiltration/diafiltration process is maintained in the desired range (i.e., about 8.5 to about 12, and preferably about 9.5 to about 10.5, for the basic mode of operation; or about 2.5 to about 4, and preferably about 2.8 to about 3.8, for the acidic mode of operation). Two methods will be described, continuous processing and batch (including semi-continuous operation) processing. It is expected that commercial processes will adopt batch or semi-continuous operation, which should be better suited to production of food-grade whey protein products. A continuous process is generally shown in FIG. 1. In either a continuous or batch process an aqueous mixture of whey protein materials is pH adjusted to solubilize whey proteins and release flavor compounds and then passed adjacent an ultrafiltration membrane which permits the lower molecular weight flavoring materials to pass through its pores along with water (the permeate), leaving the higher molecular weight whey protein materials (the retentate) to be recirculated. A portion of the retentate will be withdrawn as deflavored product, from which the whey protein materials can be recovered as needed for the ultimate end use. Water will be added to replace that lost in the permeate and to provide a constant concentration of whey protein materials in the feed stream supplied to the ultrafiltration membrane. Although not essential to the process, the process of FIG. 1 includes additional processing of the permeate to recover a portion of the water using a reverse osmosis membrane for recycling to join the retentate and fresh whey protein materials. The advantage of such a step is in reducing the amount of fresh water which must be added to the process and removed in concentrating the permeate. Of course, the pH of the whey protein materials can be kept within the desired

range by appropriate addition of a base or acid, as appropriate, to the recycled or fresh water added to the process or by direct addition of base as desired.

In a batch process, such as those described in Examples 6-8 below, a batch of whey protein material is placed in a vessel, pH adjusted, and fed to an ultrafiltration membrane. The permeate is separated and the retentate is returned to the vessel. As the process proceeds, the whey protein material is depleted in the lower molecular weight flavoring compounds and water and becomes more concentrated in the desirable whey proteins. Periodically, water is added to the retentate to dilute it and provide a carrier for the flavoring compounds which are passed through the membrane. In a semi-continuous process the water is added continuously at the rate it is being removed in the permeate. The process is continued until all of the flavoring compounds have been removed and the retentate is sufficiently deflavored to become the product, which can be further processed as required for the ultimate end use. A batch or semi-continuous process may also include the concentration of the permeate, with recycle of separated water in a similar manner as that shown in FIG. 1. The pH during the ultrafiltration/diafiltration process is maintained in the desired range (i.e., about 8.5 to about 12, and preferably about 9.5 to about 10.5, for the basic mode of operation; or about 2.5 to about 4, and preferably about 2.8 to about 3.8, for the acidic mode of operation).

The ultrafiltration membrane will be operated with a pressure differential across the membrane which assists migration of the flavoring compounds, water and other materials which are capable of passing through the pores of the membrane, while not exceeding the physical strength of the membrane. Typical average pressure for such membranes are about 50 psi (345 kPa). The trans-membrane pressure (in versus out) will be about 15 psi (103 kPa). Of course, these pressures could be varied based on the membrane's specifications and other operational concerns. The flow rate of the feed stream will provide sufficient residence time for significant permeate removal, but also will be high enough to provide turbulence so that the access

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of the feed stream to the membrane pores will not be hindered by solid deposits on the membrane walls. One skilled in the art will understand that suitable operating parameters will be determined by experience with the materials being separated.

5 In a preferred embodiment (i.e., the basic mode of operation), the present invention provides a method for preparing decaffeinated whey protein material, said method comprising: (a) providing an aqueous composition of a whey protein material containing soluble whey proteins, flavoring compounds, and insoluble materials; (b) solubilizing the whey proteins by adjusting the
10 aqueous composition of (a) to a pH in the range of about 8.5 to about 12 and releasing the flavoring compounds; (c) removing the insoluble materials from the pH-adjusted aqueous composition of (b) to obtain a treated aqueous composition; (d) passing the treated aqueous composition of (c) adjacent an ultrafiltration membrane having a molecular weight cutoff up to about 50,000
15 Daltons, while maintaining the pH in the range of about 8.5 to about 12, under suitable ultrafiltration conditions wherein the flavor compounds pass through the membrane, thereby decaffeinating the whey protein material and retaining substantially all of the solubilized whey proteins; and (e) recovering the solubilized whey proteins retained by the ultrafiltration membrane to obtain
20 the decaffeinated whey protein material.

 In another preferred embodiment (i.e., the acid mode of operation), the present invention provides a method for preparing decaffeinated whey protein material, said method comprising: (a) providing an aqueous composition of a whey protein material containing soluble whey proteins, flavoring compounds,
25 and insoluble materials; (b) solubilizing the whey proteins by adjusting the aqueous composition of (a) to a pH in the range of about 2.5 to about 4 and releasing the flavoring compounds; (c) removing the insoluble materials from the pH-adjusted aqueous composition of (b) to obtain a treated aqueous composition; (d) passing the treated aqueous composition of (c) adjacent an
30 ultrafiltration membrane having a molecular weight cutoff up to about 50,000 Daltons, while maintaining the pH in the range of about 2.5 to about 4, under suitable ultrafiltration conditions wherein the flavor compounds pass through

the membrane, thereby deflavoring the whey protein material and retaining substantially all of the solubilized whey proteins; and (e) recovering the solubilized whey proteins retained by the ultrafiltration membrane to obtain the deflavored whey protein material.

5 These preferred embodiments are illustrated in FIG. 2 wherein the pH of an aqueous solution of whey protein is adjusted to either (1) about 8.5 to about 12 for the basic mode of operation or (2) about 2.5 to about 4 for the acidic mode of operation. The pH-adjusted aqueous solution is then treated to remove insoluble materials. Any conventional technique (e.g., filtration,
10 decantation, centrifugation, and the like) can be used. Preferably, the insoluble material is removed by centrifugation. Commercial available continuous centrifugation units are ideally suited for this separation in a semi-batch or continuous type operation. In an especially preferred embodiment, the pH-adjusted aqueous is subjected to the removal technique (e.g.,
15 centrifugation) at least twice in order facilitate or more complete removal of insoluble materials. The treated supernatant is then subjected to ultrafiltration, preferably combined with diafiltration, in order to remove the flavor components normally associated with whey and whey protein compositions. During ultrafiltration, the pH of the whey protein material
20 should be maintained in the same range as used in the initial adjustment of the aqueous solution. The deflavored whey protein solution may be used directly or it may be converted to a solid form if desired. Any conventional technique for removing water can be used. Generally, spray or freeze drying techniques are preferred.

25 **Deflavored Whey Protein Products.** The deflavored whey protein materials prepared by the present methods are ideally suited for use in dairy and non-dairy beverages, smoothies, health drinks, cheeses, cheese analogs, dairy and non-dairy yogurts, meat and meat analog products, cereals, baked products, snacks, and the like. Generally, such food products
30 may contain up to about 40 percent deflavored whey proteins without significantly impacting organoleptic properties. More preferably, such food products contain about 10 to about 30 percent deflavored whey proteins.

Using the deflavored whey protein of this invention it is now possible to incorporate whey protein in conventional food products at sufficiently high levels (generally sufficient to provide about 2.5 to about 20 g whey protein per single serving size (generally about 25 to about 100 g for solid food products and about 100 to about 300 ml for liquid food products)) to provide health benefits of whey protein. Prior to this invention, incorporation whey proteins at such levels generally resulted in significant off-flavors and, therefore, only very limited acceptance by consumers.

Unless noted otherwise, all percentages are by weight. All references cited herein are incorporated by reference.

EXAMPLE 1. Whey protein concentrate (30 lbs. of WPC34 (34 percent protein; Leprino Co., Denver, CO) was hydrated with water (170 lbs.) in a mixing tank with vigorous mixing at a temperature of about 120°F. Once hydration was complete (generally within about 10 minutes), the pH was adjusted to 9 using 1 N NaOH. The pH-adjusted solution was then diafiltered through a ultrafiltration membrane (spiral wound type with 10,000 molecular weight cut-off). Diafiltration was continued for an equivalent of 5 wash cycles (each wash defined as the amount of permeate collected equal to one-half of the initial batch size). The pH was maintained at about 9 during the ultrafiltration/diafiltration process. Once diafiltration was completed, solids in the retentate was concentrated to about 20 percent and citric acid (1%) was added to adjust the pH to 6.0. The resulting slurry was freeze dried to obtain a solid deflavored whey protein material. The deflavored whey protein material was found to contain about 2.8 percent ash, about 10.2 percent carbohydrates, about 8.4 percent fat, about 2.2 percent moisture, and about 76.3 percent protein.

Slurries of the defavored whey protein material and several whey protein-containing control samples were prepared by hydrating the solid materials in water at about 100°F for about 30 minutes. The samples generally contained about 8 g whey protein per 100 g solution. Controls included untreated WPC34 as well as untreated AMP 800 (80 percent

protein; Leprino Co., Denver, CO). The AMP 800 contained approximately the same protein level as the decaffeinated whey protein inventive sample.

Based on an evaluation using a trained taste panel, the decaffeinated whey protein sample had the best overall taste, followed by the AMP 800 control, with the WPC34 (untreated) having the most off flavors. More specifically, the lactose, milky, and animal flavors normally associated with whey proteins (and detected in both control samples) were essentially eliminated in the inventive sample.

EXAMPLE 2. The decaffeinated whey protein sample prepared in

Example 1 was used to prepare a high protein beverage. The following formulation was prepared: 87.3 percent water, 7.0 percent decaffeinated whey protein, 2.5 percent salt, 2.5 percent sugar, and 0.2 percent peach flavors. The dry components were first blended and then hydrated in the water using an overhead mixer. Once hydration was complete, the flavor component was added. Stabilizers such as pectin and carrageenan could be added if desired to adjust final product viscosity to the desired level. Sugar could be replaced to high corn syrup or other natural or artificial sweeteners if desired. Based on taste evaluation using a trained taste panel, the beverage was considered excellent with regard to overall mouthfeel and flavor with no off-flavors detected. The resulting beverage delivers about 13 g whey protein per 250 ml serving size.

EXAMPLE 3. This example illustrates the preparation of decaffeinated whey protein from sweet whey. A clarified sweet whey (also known as rennet whey or cheese whey) was obtained from a cheese making process. The sweet whey contained about 12 percent protein, about 78 percent carbohydrates, and about 1.1 percent fat on a dry basis (remainder mainly ash). The sweet whey had been clarified using centrifugation to remove fat, cheese fines, and caseins, thereby significantly reducing fouling and increasing flux rates in the subsequent ultrafiltration step. About 700 pounds of the sweet whey was heated to about 120°F in a jacketed reactor; the pH was then adjusted to about 7.5 with the addition of 1N NaOH. The alkalized

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5 whey was then continuously concentrated using ultrafiltration/diafiltration as in Example 1 using a UF spiral membrane with a 10000 molecular weight cutoff. Retentate was recirculated and maintained at about 5 percent solids. Once the solids in the permeate reached about 50 percent of the original solids in the retentate, the pH was readjusted to about 9 using 1N NaOH; the pH was maintained at about the same level throughout the remainder of the UF/DA process. Diafiltration was continued at a constant solids level by the addition of reverse osmosis water at the same rate as removal of permeate until the ratio of solids in the permeate to solids in the retentate reached about 0.1 to about 0.15. The retentate was concentrated to about 10 percent solids, collected, neutralized to pH 6.5 using citric acid (1 percent), and then pasteurized (165°F for 30 seconds). The pasteurized product was then refrigerated before evaluation. The resulting product contained about 68 percent protein, about 16 percent carbohydrates, and about 6.3 percent fat on a dry basis. The flavor was bland with no off-flavors making it ideal for incorporation into beverages and other food products.

20 **Example 4.** This example illustrates the preparation of deffavored whey protein from acid whey. Clarified acid whey as concentrated to about 14 percent solids and about 50 percent protein. Clarification was by carried out by centrifugation as in Example 3. Concentrated acid whey (200 pounds) was diluted with about 200 pounds reverse osmosis water and then heated to about 120°F in a jacketed tank. The pH of the slurry (Initially about 4.7) was adjusted to about 9 by slowly adding 1 N NaOH. The alkalized whey was then continuously concentrated using ultrafiltration/diafiltration as in Example 1 using a UF spiral membrane with a 10000 molecular weight cutoff. Reverse osmosis water was added as needed to maintain solids at about 4 to about 7 percent. The pH was maintained at about 9 throughout the UF/DA process. Diafiltration was continued at a constant solids level by the addition of reverse osmosis water at the same rate as removal of permeate until the ratio of solids in the permeate to solids in the retentate reached about 0.1 to about 0.15. The retentate was concentrated to about 14 percent solids, collected, neutralized to pH 6.5 using citric acid (1 percent), and then pasteurized

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(165°F for 30 seconds). The pasteurized product was then refrigerated before evaluation. The resulting product contained about 80 percent protein, about 9 percent carbohydrates, and about 4 percent fat on a dry basis (remainder mainly ash). The flavor was bland with no off-flavors making it ideal for incorporation into beverages and other food products. A portion of the product was spray dried for further use and evaluation.

Example 5. This examples illustrates the preparation of a high-acid fruit juice beverage containing about 8 g protein per single serving size (about 240 ml) derived from the spray dried deflavored acid whey of Example 4. The beverage was prepared using the following formulation:

Ingredient	Amount (%)
Water	79.3
Deflavored Acid Whey Powder	4.1
Evaporated Cane Juice	9.0
Alginate	0.3
Citric Acid	0.1
Malic Acid	0.2
Phosphoric Acid (80%)	0.1
Ascorbic Acid	0.03
Strawberry Juice Concentrate (85 Prbx)	0.4
White Grape Juice Concentrate (68 Prbx)	6.4
Strawberry Flavor	0.2
Coloring (red)	0.002

The deflavored acid whey powder was hydrated in a portion of the water (at about 160°F). The remainder of the water was to about 160°F and the alginate and evaporated cane juice added with high agitation. The hydrated whey powder mixture was then added, followed by the various acids, juice concentrates, and remaining ingredients. The mixture was homogenized using a high speed mixer for about 5 minutes. The resulting mixture, which had good flavor, had a pH of about 4.1. Lower pH values (e.g., about 3.5 to

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about 4) increases stability. Likewise, stabilizers (e.g., pectin, poly-glutamic acid, and the like) can be added at relatively low values (e.g., about 0.3 to about 0.4 percent) to increase stability if desired.

Example 6. This examples illustrates the preparation of a low acid chocolate beverage containing about 8 g protein per single serving size (about 240 ml) derived from the decaffeinated acid whey of Example 4. The beverage was prepared using the following formulation:

Ingredient	Amount (%)
Filtered Water	83.4
Decaffeinated Acid Whey Powder	4.1
Potassium Citrate	0.4
Sea Salt	0.03
Microcrystalline Cellulose	0.25
Evaporated Cane Juice	8.5
Cocoa	1.5
Cream	1.5
Vanilla Flavor	0.3

The filtered water was divided into two portions of about the same size. The decaffeinated acid whey powder, about 60 percent of the evaporated cane juice, the microcrystalline cellulose, potassium citrate, and the sea salt were preblended and then added to the first water portion with high agitation to form a first slurry; the cream was then added to the first slurry. The cocoa and the remainder of the evaporated can juice were then preblended and then added to the second water portion with high agitation to form a second slurry. The second slurry was then mixed with the first slurry and then homogenized in a two stage homogenizer (2500/500 psi). After pasteurization at about 195°F for about 5 to about 10 seconds, the beverage was sealed in heat stable containers and then chilled to about 45°F. Evaluation with a trained test panel indicated that the product had good mouthfeel and texture, excellent cocoa aroma, and pleasant chocolate flavor

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with no off-flavors or undesirable aftertaste. The vanilla flavor was apparently at too low a concentration to be detected; it may be desirable to increase its level.

Example 7. This example illustrates the acidic mode of operation for the deflavoring process. Clarified concentrated acid whey (300L lbs; WPC 50 as used in Example 4; initial pH 4.7) was diluted with 100 lbs of deionized water in a jacketed tank with overhead mixing. The mixture was heated to about 120°F by circulating hot water through the jacket. The pH was then adjusted to 3.2 by slowly adding 10% phosphoric acid. The pH-adjusted whey solution was allowed to equilibrate for about 10 minutes and then was continuously concentrated using ultrafiltration/diafiltration as in Example 1 using a UF spiral membrane with a 10000 molecular weight cutoff. Continuous ultrafiltration/diafiltration was carried out for an equivalent of 6 wash cycles at which time the amount of solids in permeate was close to zero. The pH was maintained at about 3.2 throughout the UF/DA process. The retentate was then concentrated to a solids content of about 12 percent. The product was then batched pasteurized at 165°F for 5 minutes. The pasteurized product was then refrigerated before evaluation. The resulting product contained about 80 percent protein, about 5 to about 10 percent carbohydrates, and about 1 to about 3 percent fat on a dry basis (remainder mainly ash). The flavor was bland with no off-flavors making it ideal for incorporation into beverages and other food products.

Example 8. This example illustrates the preparation of a mixed berry, high acid, whey-containing beverage using the deflavored whey protein obtained in Example 7. The following formulation was used:

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Ingredient	Amount (%)
Deionized Water	38.8
Deionized Acid Whey (liquid)	45.6
Sucrose	11.1
Strawberry Juice Conc. (65 Brx)	0.7
White Grape Juice Conc. (68 Brx)	1.8
Clarified Orange Juice Conc. (58 Brx)	1.0
Sour Cheery Juice Conc. (68 Brx)	0.2
Red Raspberry Juice Conc. (68 Brx)	0.2
Coloring	0.001
Sodium Citrate	0.05
Natural Cherry Flavor	0.05
Natural Mixed Berry Flavor	0.3
Natural Blueberry Flavor	0.04
Vitamin Mixture	0.02
Other Natural Flavors	0.1

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The deionized water at 150°F and the deionized whey were mixed together. The remaining ingredients were then added in the following order with blending: dry ingredients (sucrose, sodium citrate, vitamin premix, coloring), juice concentrates, and flavors. The resulting beverage was filled into sterile 1-liter bottles. The product was preheated to 170°F in preheat exchangers and then pasteurized at a minimum temperature of 230°F and a minimum time of 2 seconds. The product was homogenized at 170°F, then cooled to about 40°F, and filled into individual bottles.

A beverage prepared in such a manner would be expected to have the following nutritional characteristics per single serving size of about 258g; Calories: 150; Total Fat: 0g; Cholesterol: 0mg; Sodium: 30 mg; Total Carbohydrate: 26g; Dietary Fiber: 0g; Sugars: 24g; Protein 10g; Vitamin A: 30%; Vitamin C: 30%; Calcium 15%; Vitamin E: 30%. Such a beverage would be considered fat free, saturated fat free, and cholesterol free as well as being an excellent source of protein and vitamins A, C, and E.

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The finished product provided excellent mouthfeel and was less astringent than similar products prepared with non-deflavored whey products. Since the deffavored whey material was prepared using the acid mode of operation, its pH was close to the desired pH of the final beverage and, therefore, it was not required to be adjusted in such a manner as to pass
5 through its isoelectric point.

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CLAIMS

1. A method for preparing deflavored whey protein material, said method comprising:

(a) preparing an aqueous composition of a whey protein material containing soluble whey proteins and flavoring compounds;

(b) adjusting the aqueous composition of (a) to either (1) a basic pH in the range of about 8.5 to about 12 or (2) an acidic pH in the range of about 2.5 to about 4, thereby releasing the flavoring compounds;

(c) passing the pH-adjusted aqueous composition of (b) adjacent an ultrafiltration membrane having a molecular weight cutoff up to about 50,000 Daltons, while maintaining the pH in the same range as adjusted in step (b), under suitable ultrafiltration conditions wherein the flavor compounds pass through the membrane, thereby deflavoring the whey protein material and retaining substantially all of the soluble whey proteins; and

(d) recovering the soluble whey proteins retained by the ultrafiltration membrane to obtain the deflavored whey protein material.

2. The method of claim 1, wherein the pH of the aqueous composition is adjusted to the basic pH in step (b).

3. The method of claim 1, wherein the pH of the aqueous composition is adjusted to the acidic pH in step (b).

4. The method of claim 1, wherein the whey protein material is at least one member of the group consisting of whey from a cheese making process, whey protein isolate, and whey protein concentrate.

5. The method of claim 1, wherein the whey protein material is treated prior to step (c) to remove any water insoluble material that may be present.

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6. The method of claim 5, wherein the water insoluble material that may be present is removed from the pH-adjusted aqueous composition using at least one centrifugation step.

7. The method of claim 5, wherein the water insoluble material that may be present is removed using at least one clarification step.

8. The method of claim 4, wherein the aqueous composition of (a) has a concentration of soy material in the range of about 1 to about 50 percent.

9. The method of claim 5, wherein the aqueous composition of (a) has a concentration of soy material in the range of about 1 to about 50 percent.

10. The method of claim 4, wherein the ultrafiltration membrane has a cutoff in the range of about 1,000 to about 50,000 Daltons.

11. The method of claim 5, wherein the ultrafiltration membrane has a cutoff in the range of about 1,000 to about 50,000 Daltons.

12. The method of claim 10, wherein the ultrafiltration membrane has a cutoff in the range of about 10,000 to about 30,000 Daltons.

13. The method of claim 11, wherein the ultrafiltration membrane has a cutoff in the range of about 10,000 to about 30,000 Daltons.

14. The method of claim 12, wherein the ultrafiltration is carried out at a temperature in the range of about 10 to about 60°C and a suitable pressure.

15. The method of claim 13, wherein the ultrafiltration is carried out at a temperature in the range of about 10 to about 60°C and a suitable pressure.

16. The method of claim 14, wherein the ultrafiltration membrane is a polymer, ceramic, or inorganic membrane.

17. The method of claim 15, wherein the ultrafiltration membrane is a polymer, ceramic, or inorganic membrane.

18. The method of claim 1, wherein the deflavored whey protein material is further treated to remove water to obtain a solid deflavored whey protein material.

19. The method of claim 6, wherein the deflavored whey protein material is further treated to remove water to obtain the deflavored whey protein material in a solid form.

20. The method of claim 9, wherein the deflavored whey protein material is further treated to remove water to obtain the deflavored whey protein material in a solid form.

21. The method of claim 1, wherein the deflavored whey protein material is spray dried to remove water to obtain the deflavored whey protein material in a solid form.

22. The method of claim 6, wherein the deflavored whey protein material is spray dried to remove water to obtain the deflavored whey protein material in a solid form.

23. The method of claim 7, wherein the deflavored whey protein material is spray dried to remove water to obtain the deflavored whey protein material in a solid form.

24. A whey-containing food product comprising a deflavored whey protein, wherein the whey-containing food product is prepared by

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incorporating the deflavored whey protein into a food product and wherein the deflavored whey protein is prepared by a process comprising:

(a) preparing an aqueous composition of a whey protein material containing soluble whey proteins and flavoring compounds;

(b) adjusting the aqueous composition of (a) to either (1) a basic pH in the range of about 8.5 to about 12 or (2) an acidic pH in the range of about 2.5 to about 4, thereby releasing the flavoring compounds;

(c) passing the pH-adjusted aqueous composition of (b) adjacent an ultrafiltration membrane having a molecular weight cutoff up to about 50,000 Daltons, while maintaining the pH in the same range as adjusted in step (b), under suitable ultrafiltration conditions wherein the flavor compounds pass through the membrane, thereby deflavoring the whey protein material and retaining substantially all of the soluble whey proteins; and

(d) recovering the soluble whey proteins retained by the ultrafiltration membrane to obtain the deflavored whey protein material

25. The whey-containing food product of claim 24, wherein the pH of the aqueous composition is adjusted to the basic pH in step (b).

26. The whey-containing food product of claim 24, wherein the pH of the aqueous composition is adjusted to the acidic pH in step (b).

27. The whey-containing food product of claim 24, wherein the food product is selected from the group consisting of dairy and non-dairy beverages, smoothies, health drinks, cheeses, cheese analogs, dairy and non-dairy yogurts, meat and meat analog products, cereals, baked products, and snacks.

28. The whey-containing food product of claim 24, wherein the whey-containing food product contains about 10 to about 30 percent deflavored whey protein.

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29. The whey-containing food product of claim 25, wherein the whey-containing food product contains about 10 to about 30 percent deflavored whey protein.

30. The whey-containing food product of claim 26, wherein the whey-containing food product contains about 10 to about 30 percent deflavored whey protein.

31. The whey-containing food product of claim 25, wherein the whey-containing food product is a beverage having a pH of greater than 5.

32. The whey-containing food product of claim 26, wherein the whey-containing food product is a beverage having a pH of less than 4.5.

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METHOD OF DEFLAVORING WHEY PROTEIN MATERIALS

ABSTRACT

Whey protein materials such as whey from cheese making processes, whey protein concentrates, and whey protein isolates, are deflavored by adjusting the pH of an aqueous composition of such whey protein materials to about 8 to about 12 to solubilize the whey proteins and to release the flavoring compounds and thereafter passing the pH-adjusted composition to an ultrafiltration membrane having a molecular weight cutoff up to about 50,000 Daltons under conditions at which the flavoring compounds pass through the membrane, leaving the retained whey protein material with improved flavor.

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**DECLARATION
FOR UTILITY OR DESIGN
PATENT APPLICATION**

	)	Attorney Docket No.:	1410/77018
	)	Customer No.:	22242
	)		
	)	First Named Inventor:	AKASHE et al.
☒ Declaration Submitted With	)	Application Number:	N/A
Initial Filing	)		
☐ Declaration Submitted After	)	Filing Date:	HEREWITH
Initial Filing	)	Art Unit:	N/A
	)	Examiner Name:	Not yet assigned

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

METHOD OF DEFLAVORING WHEY PROTEIN

(Title of Invention)

the specification of which:

☒ is attached hereto, or

☐ was filed by an authorized person on my behalf on _____ as United States Application Number _____ or PCT International Application Number _____, and was amended on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT International application which designated at least one country other than the United States of America, listed below, and I have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or any PCT international application, on this invention filed by me or my legal representatives or assigns and having a filing date before that of the application on which priority is claimed:

for 102

Prior Foreign Application Number(s)	Country	Foreign Filing Date	Priority Not Claimed
			☐
			☐
			☐

☐ Additional foreign application numbers are listed on a supplemental priority data sheet attached hereto.

I hereby claim the benefit under Title 35, United States Code, § 119(e) of any United States provisional application(s) listed below:

Provisional Application Number(s)	Provisional Application Filing Date
--	--

☐ Additional provisional application numbers are listed on a supplemental priority data sheet attached hereto.

I hereby claim the benefit under Title 35, United States Code, § 120, of any prior United States application(s), or under § 365(c) of any PCT international application(s) designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT international application(s) in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose all information known by me to be material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56, which became available between the filing date of the prior application(s) and the national or PCT international filing date of this application:

Prior U.S. Application Number	Prior PCT International Application Number	Filing Date of U.S. or PCT International Application	Patent Number (if applicable)
--	---	---	--

☐ Additional U.S. or PCT international application numbers are listed on a supplemental priority data sheet attached hereto.

As a named inventor, I hereby appoint Debbie K. Wright, Registration No. 33,817; Thomas A. Marcoux, Registration No. 29,826; Valerie Calloway, Registration No. 40,546; and the practitioners associated with Customer Number 22242, with full power of substitution and revocation, to prosecute this application and to transact all business in the United States Patent and Trademark Office connected therewith, and request that all correspondence and

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157 51

Attorney Docket No. 1410/77016

telephone calls in respect to this application be directed to FITCH, EVEN, TABIN & FLANNERY, Suite 1600, 120 South LaSalle Street, Chicago, Illinois 60603-3408, Telephone No. (312) 577-7000, Facsimile No. (312) 577-7007, CUSTOMER NUMBER 22242.

I hereby declare that all statements made herein of my own knowledge are true, and that all statements made herein on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity or enforceability of the application or any patent issued thereon.

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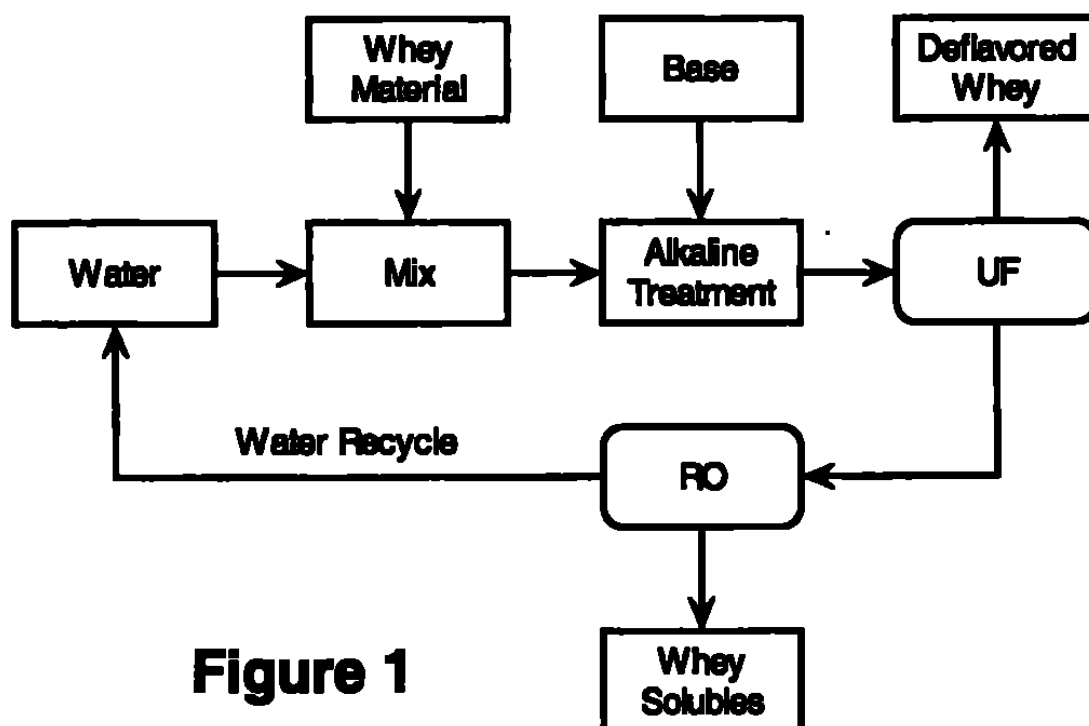


Figure 1

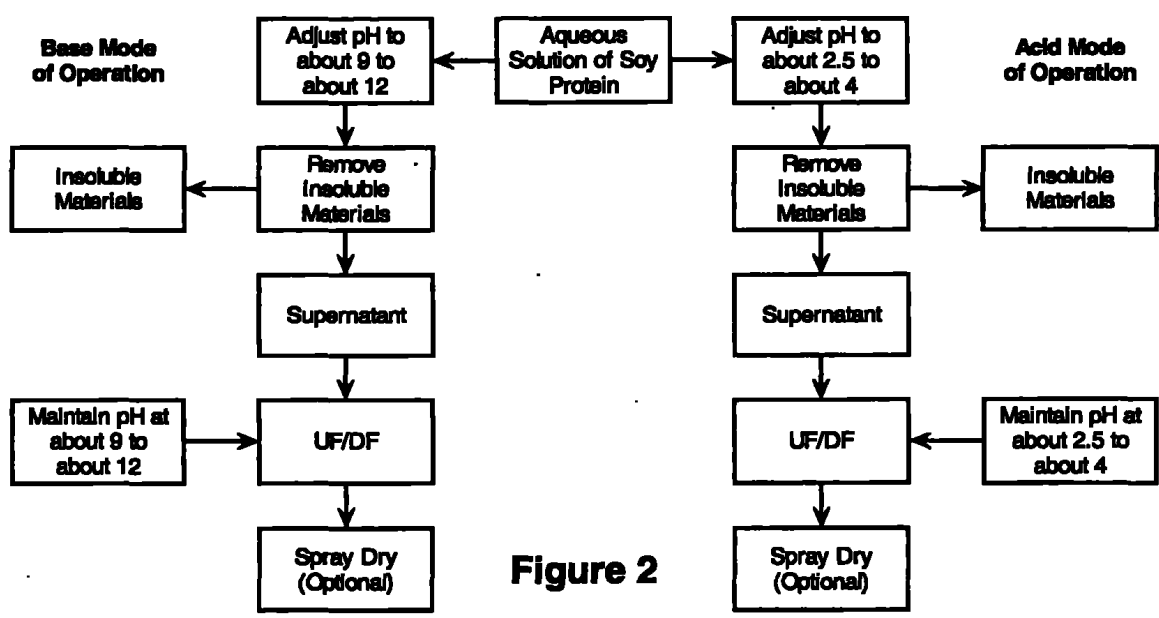


Figure 2

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