Industria y Comercio
SUPERINTENDENCIA

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
No. 08-083008--00006-0000
Fecha: 2010-04-27 10:58:03 Dep: 2020 DIR.NUEVASCR
Tra. 11 PATENTEIYII Eve: 379 FASENACIONALII
Act. 400 OPOSIOBSERVTER Folios: 41

FORMULARIO ÚNICO DE OPOSICIÓN

① OPOSICIÓN A :

- [X] Patente de Invención.
- [ ] Patente de Modelo de Utilidad.
- [ ] Diseño Industrial.
- [ ] Esquema de Trazado para Circuitos Integrados.
- [ ] Marcas de Productos o Servicios.
- [ ] Marca Colectiva.
- [ ] Marca de Certificación.
- [ ] Lema Comercial.

② SOLICITUD PUBLICADA

Número del expediente: 08 083.008
Solicitante: MISSION PHARMACAL COMPANY, STERLING FOODS LTDA
Apoderado: DILIA MARIA RODRIGUEZ D` ALEMAN
Título de la nueva creación o denominación del signo: PRODUCTO ALIMENTICIO ENRIQUECIDO CON CALCIO
Gaceta: 612

③ OPOSICIÓN PRESENTADA POR:

SOLICITANTE

Nombre: PROCAPS S.A.
Dirección: CALLE 80 No. 78b-201
Nacionalidad o Domicilio: COLOMBIA
Lugar de Constitución: BARRANQUILLA
Teléfono: 371 99 00 Fax: 373 08 18
E-mail:

IDENTIFICACIÓN
C.C. [ ] NIT [X]
C.E. [ ] Otro [ ]
Cual
Número 890.106.527-5

REPRESENTANTE O APODERADO

Nombre: CIELO ANGELA PEÑA RODRIGUEZ
Dirección: CARRERA 13 No. 119-95
Teléfono: (571)213 12 13 Fax: (571)612 19 79
E-mail: negocios@optimum-co.com

IDENTIFICACIÓN
C.C. [X] NIT [ ]
C.E. [ ] Otro [ ]
Cual
Número 51.940.252 TP 82701

④ FUNDAMENTOS DE LA OPOSICIÓN:

Art.14, 16, 18, 20 y 30 de la Decisión 486 de la CAN

Causal: No cumple con los requisitos de patentabilidad.

Signo opositor:

Justificación:

La presente solicitud no cumple con los requisitos de patentabilidad dado que lo revelado, ya se encontraba en el estado de la técnica o es posible derivarlo de la misma tal y como se demostrará en el capítulo "Fundamento de Hechos".

2

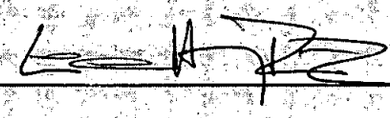
⑤ **Comprobante de pago No.** _____ **Fecha** _____

⑥ **ANEXOS**

- [x] Comprobante de pago de la tasa.
- [x] Poderes, si fuere el caso.
- [x] Pruebas de los fundamentos de la oposición.
- [ ] Documentos que acrediten el legítimo interés, si fuere el caso.
- [x] Documento que acredita la existencia y representación legal de las partes, cuando sean personas jurídicas.

⑦

NOMBRE: CIELO ANGELA PEÑA RODRIGUEZ

FIRMA: 

C.C. 51.940.252 **TP.** 82701

Instrucciones para el diligenciamiento del presente formulario, ver reverso de esta página.

Optimum

Derecho de la Competencia, Propiedad Industrial y Comercio Exterior

Señora Jefe,

DIVISIÓN DE NUEVAS CREACIONES

Superintendencia de Industria y Comercio

E. S. D.

Referencia	: Expediente N° 08 083008
Asunto	: Oposición a la solicitud de patente de Invención titulada "PRODUCTO ALIMENTICIO ENRIQUECIDO CON CALCIO"
Solicitante	: MISSION PHARMACAL CO.
Opositora	: PROCAPS S.A.

Publicada en la Gaceta de la Propiedad Industrial

N° 612 del 29 de enero de 2010

Yo, *CIELO ANGELA PEÑA RODRIGUEZ*, abogada en ejercicio, identificada como aparece al pie de su firma, con tarjeta profesional No. *82701* del Consejo Superior de la Judicatura, en mi condición de apoderada de la sociedad **PROCAPS *S.A.*** conformada bajo las leyes de la República de Colombia y domiciliada en la ciudad de Barranquilla, Colombia, atentamente manifiesto que estando dentro del término de ley y por orden expresa de mi representada, presento oposición a la concesión del registro de la Patente de Invención titulada **"PRODUCTO ALIMENTICIO ENRIQUECIDO CON CALCIO"**, cuyo titular es **MISSION PHARMACAL CO.**, y pido a Usted declarar fundada esta oposición y en consecuencia negar el registro de la patente solicitada.

Carrera 13 N° 119-95, Of. 104 Tel. (571) 2 13 12 13 - 6 20 50 21 - Fax. (571) 6 12 19 79
E-mail: negocios@optimum-co.com - www.optimum-co.com
Bogotá Colombia

1. INTERÉS PARA ACTUAR

Conforme a lo dispuesto en el artículo 42 de la Decisión 486 de 2000 de la CAN, estando dentro del término estipulado por la ley, manifiesto el legítimo interés de nuestra representada para oponerse al registro de la patente de la referencia, atendiendo a que la solicitud pretendida no cumple con los requisitos dispuestos en la ley como se analizará más adelante y de ser concedida afectaría directamente la comercialización de algunos productos de mi representada, que contienen, en particular **VITAMINA D3.** Por lo tanto, solicito respetuosamente a ese Despacho se sirva tener en cuenta la presente oposición dentro del trámite administrativo que se surte en esta Superintendencia.

2. FUNDAMENTOS DE HECHOS

2.1. La solicitud objeto de la presente fue presentada el 08 de agosto de 2008 reivindicando prioridad bajo la solicitud US20060329905 del 2006-01-11; y fue publicada en Colombia, en la Gaceta de la Propiedad Industrial **N° 612 del 29 de enero de 2010**, cuya publicación internacional corresponde a la WO2007/081659 del 19/07/2007.

2.2. La solicitud colombiana **08 083008** objeto de la presente oposición, se refiere a un producto alimenticio enriquecido, tal como un producto alimenticio masticable enriquecido con calcio en el que al menos una pieza del producto alimenticio proporciona la (RDI) recomendada diaria de calcio elemental. El producto alimenticio enriquecido tiene un gusto, una textura y una sensación en

la boca sustancialmente similares a un producto alimenticio no enriquecido. El producto alimenticio también puede proporcionar vitamina D3 en un intervalo de 100-2400 UI y opcionalmente puede cubrirse con un recubrimiento aromatizado.

2.3. Con base en el artículo 14 de la Decisión 486 de la CAN que consagra lo siguiente: *"Los países miembros otorgarán patentes para las invenciones, sean de producto o procedimiento, en todos los campos de la tecnología, siempre que sean nuevas, tengan nivel inventivo y sean susceptibles de aplicación industrial"*. Así las cosas, las invenciones objeto de patente deben ser nuevas y tener nivel inventivo. Bien, pues antes de la fecha de prioridad de esta solicitud se encuentran los siguientes documentos que afectan la patentabilidad de la presente solicitud:

- D1: US6723358 publicada el 20 de abril de 2004.
- D2: US6086927 publicada el 11 de julio de 2000.

2.4.Ahora bien, el documento D1, particularmente en la columna 9, líneas 11 a 24, columna 13, líneas 43 a 62 y en la columna 17, líneas 33 a 45 revela alimentos de calcio enriquecido masticables, que contienen sabor, particularmente sabor a chocolate, aceites y grasas como las del grupo omega, antioxidantes, minerales como magnesio, manganeso, zinc, hierro y fosforo, vitaminas como la vitamina A, el grupo vitaminas B, vitamina C, d y k, como se puede ver en la columna 9, líneas 10 a 24 y continúa hasta la columna 25 línea 56 con los compuestos farmacéuticos fenitoina, ibuprofeno y aspirina; alimento enriquecido que puede o no ser horneado o cocido.

2.5.De igual manera, se muestra en el documento D2 particularmente en las columnas 1 y un producto alimenticio enriquecido con calcio que comprende: un producto alimenticio en el que al menos una pieza del producto proporciona la (RDI) ración diaria recomendada de calcio; en el que el producto alimenticio enriquecido con calcio tiene una sensación en la boca, textura y sabor muy similares a un producto alimenticio no enriquecido.

2.6.Considerando los documentos D1 y D2 el estado de la técnica más cercano a la invención tenemos que no es posible reconocer novedad ya que un producto alimenticio enriquecido, tal como un producto alimenticio masticable enriquecido con calcio en el que al menos una pieza del producto alimenticio proporciona la (RDI) recomendada diaria de calcio elemental. El producto alimenticio enriquecido tiene un gusto, una textura y una sensación en la boca sustancialmente similares a un producto alimenticio no enriquecido; producto alimenticio que también puede proporcionar vitamina D3 y que opcionalmente puede cubrirse con un recubrimiento aromatizado, eran ampliamente conocidas a la fecha de prioridad. Por lo tanto, la solicitud objeto de la presente oposición no puede comprender patente toda vez que no cumple con el requisito más importante, la NOVEDAD, dado que lo que pretende ya es ampliamente conocido en el arte anterior.

2.7.Tomando los documentos D1 y D2, no es posible reconocer altura inventiva, ya que sería obvio para un experto en la materia a partir de los documentos D1, y D2 apoyado en el hecho de usar vitaminas, saborizantes y compuestos farmacéuticos, concebir un producto alimenticio masticable enriquecido con calcio en el que al menos una pieza del producto alimenticio proporciona la (RDI) recomendada diaria de calcio elemental, que tiene un gusto, una textura y una sensación en la boca sustancialmente similares a un producto alimenticio no

enriquecido; y producto alimenticio que también puede proporcionar vitamina D3 y que opcionalmente puede cubrirse con un recubrimiento aromatizado. Así entonces las reivindicaciones de la solicitud objeto de la presente oposición, carecen de altura inventiva.

2.8. En consecuencia y de acuerdo a lo descrito anteriormente, se deriva que la solicitud colombiana **08 083008** no puede pretender un monopolio por patente toda vez que lo que pretende no cumple con dos de los tres requisitos necesarios para su protección.

2.9.Que por consiguiente, respecto a la solicitud pretendida se tiene que:

2.9.1. CARECE DE NOVEDAD: Según la decisión 486 de la Comunidad Andina de Naciones, Artículo 16.- *"Una invención se considerará nueva cuando no está comprendida en el estado de la técnica. El estado de la técnica comprenderá todo lo que haya sido accesible al público por una descripción escrita u oral, utilización, comercialización o cualquier otro medio antes de la fecha de presentación de la solicitud de patente o, en su caso, de la prioridad reconocida.".* A la fecha de prioridad, 11 de enero de 2006 ya era conocido, pues como se demostró, era parte del arte anterior con base en los documentos D1 y D2", un producto alimenticio masticable enriquecido con calcio en el que al menos una pieza del producto alimenticio proporciona la (RDI) recomendada diaria de calcio elemental. El producto alimenticio enriquecido tiene un gusto, una textura y una sensación en la boca sustancialmente similares a un producto alimenticio no enriquecido; producto alimenticio que también puede proporcionar vitamina D3 y que

opcionalmente puede cubrirse con un recubrimiento aromatizado, sobre el cual busca protección en el capítulo reivindicatorio.

2.9.2. CARENCIA DE ALTURA INVENTIVA. Según la Decisión 486 de la Comunidad Andina de Naciones, Artículo 18.- *"Se considerará que una invención tiene nivel inventivo, si para una persona del oficio normalmente versada en la materia técnica correspondiente, esa invención no hubiese resultado obvia ni se hubiese derivado de manera evidente del estado de la técnica."*. A la fecha de prioridad, 11 de enero de 2006, a partir del documento D1 por si solo, o por la combinación de D1 y D2, puede llegar de manera evidente a un producto alimenticio masticable enriquecido con calcio, que puede proporcionar vitamina D3 y que opcionalmente puede cubrirse con un recubrimiento aromatizado, sobre el cual busca protección en el capítulo reivindicatorio.

2.10. Dado lo anterior, es posible deducir que la solicitud y lo reivindicado no es objeto de patentabilidad pues es contrario a lo que dicta la Decisión 486 de la Comunidad Andina de Naciones, Artículo 14. *"Los Países Miembros otorgarán patentes para las invenciones, sean de producto o de procedimiento, en todos los campos de la tecnología, siempre que sean nuevas, tengan nivel inventivo y sean susceptibles de aplicación industrial."*. La solicitud pretendida no cumple con dos de los tres requisitos de patentabilidad.

2.11. Por todo lo anterior, atentamente solicito a usted que se sirva declarar fundada la presente oposición y en consecuencia negar el registro a la solicitud de patente titulada **"PRODUCTO ALIMENTICIO ENRIQUECIDO CON CALCIO"**, cuyo titular es **MISSION PHARMACAL CO.**, solicitud Colombiana que

apareció publicada en la Gaceta de la Propiedad Industrial **N° 612 del 29 de enero de 2010.**

3. PRUEBAS

De conformidad con lo dispuesto por las normas vigentes, solicito a usted tener como prueba en este asunto los siguientes:

- D1: US6723358 publicada el 20 de abril de 2004.
- D2: US6086927 publicada el 11 de julio de 2000.

4. FUNDAMENTO DE DERECHO

Fundamento esta observación en las normas legales siguientes:

4.1. Numeral 1 del artículo 586 del Código de Comercio.

4.2. Artículo 42 de la Decisión 486 de la Comisión del Acuerdo de Cartagena.

4.3. En las demás normas concordantes.

5. ANEXOS

Para los efectos correspondientes anexo al presente escrito los siguientes documentos:

1. Poder para actuar en el presente.
2. Documento que acredita al representante legal de **PROCAPS S.A.**
3. Recibo de caja del respectivo pago de tasa.
4. Copias de los documentos:

- D1: US6723358 publicada el 20 de abril de 2004.
- D2: US6086927 publicada el 11 de julio de 2000.

Carrera 13 N° 119-95, Of. 104 Tel. (571) 2 13 12 13 - 6 20 50 21 - Fax. (571) 6 12 19 79
E-mail: negocios@optimum-co.com - www.optimum-co.com
Bogotá Colombia

Optimum

Derecho de la Competencia, Propiedad Industrial y Comercio Exterior

6. NOTIFICACIONES

Para efectos de notificaciones que por disposición del artículo 50 del Decreto 01 de 1984 debe hacer su Despacho, informo que mi representada y la suscrita recibiremos notificaciones en la Secretaría de su Despacho o en mis oficinas situadas en la Carrera 13 N° 119-95, Of. 104 de esta ciudad de Bogotá.

Señor Superintendente,

CIELO ANGELA PEÑA RODRIGUEZ
C.C. 51'940.252 de Bogotá
T.P.A. 82701 del C.S. de la J.

12

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO

NIT : 800176089-2

RECIBO OFICIAL DE CAJA : 10 - 32,631
FECHA : ABRIL 27 DE 2010

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO

No. 08-083008- -00006-0000

Fecha: 2010-04-27 10:58:03 Dep. 2020 DIR.NUEVASCR
Tra. 11 PATENTEIYII Eve: 379 FASENACIONALII
Act. 400 OPOSIOBSERVTER Folios: 41

***** C O N S I G N A C I O N *****

DEPOSITANTE	TIPO PAGO	BANCO	CUENTA	No. PAGO	FECHA PGO	Vr. PAGO
LAB. PROCAPS	CONSIGNACION	BANCO DE BOGOTA	062754387	174684996	07/04/2010	590,000.00
LAB. PROCAPS	CONSIGNACION	BANCO DE BOGOTA	062754387	174685000	07/04/2010	1,475,000.00

***** C O N C E P T O *****

CANT.	RENTISTICO	CONCEPTO	TOTAL CONCEPTO
1	50005-01-04 PRESENTACION DE OBSERVACIONES	12 MARCAS Y LEMAS COMERCIALES	295,000.00
		TOTAL : $	295,000.00

SON: DOSCIENTOS NOVENTA Y CINCO MIL PESOS

RESPONSABLE : _____

RECIBO DE CAJA APLICADO AL EXPEDIENTE No._____

PROCAPS

PODER ESPECIAL

Yo, **WALTER TANAKA TATEKAWA,** mayor de edad y vecino de la ciudad de Barranquilla (Colombia), identificado como aparece al pie de mi firma, quien en el presente documento actúa en calidad de Representante Legal de la sociedad **PROCAPS S.A.** sociedad legalmente constituida y domiciliada en la ciudad de Barranquilla (Colombia), por el presente documento otorgo a la doctora **CIELO ANGELA PEÑA RODRIGUEZ,** abogada en ejercicio, identificada como aparece al pie de su firma, con tarjeta profesional No. 82701 **del C.S. de la J.**, poder especial, amplio y suficiente, para formular oposiciones u observaciones a la solicitud de patente No. **08 083.008,** titulada **"PRODUCTO ALIMENTICIO ENRIQUECIDO CON CALCIO",** cuyo titular es **MISSION PHARMACAL CO,** publicada en la gaceta No. gaceta No. 612 del 29 de enero de 2010.

Nuestro apoderado queda facultado, de manera expresa, para realizar todo tipo de actos necesarios para llevar a cabo el trámite de la oposición, incluyendo, sin limitación, preparar y presentar solicitudes, hacer declaraciones; pagar tasas, contribuciones e impuestos, recabar los títulos o certificados. Asimismo quedan autorizados para impugnar administrativa todo lo que fuere resuelto en el expediente a que se hace expresa referencia en el presente poder, con todas las facultades generales y específicas que fuera requerido para ella.

De la misma manera, además de las facultades que le confiere la naturaleza del presente mandato, se encuentran facultados para recibir, desistir, transigir, sustituir, y reasumir el presente poder, incoar acciones, interponer recursos y demás facultades conferidas por la ley.

Dado y Firmado en Barranquilla (Colombia) a los 12 días del mes de abril de 2010.

PROCAPS S.A.

C.C. N° 16'251.923
NIT: 890.106.527-5
Representante Legal

Acepto:

CIELO ANGELA PEÑA RODRIGUEZ
C.C. 51'940.252 de Bogotá
T.P.A. 82701 del C.S. de la J.

NOTARIA TERCERA DEL CIRCULO DE BARRANQUILLA

DILIGENCIA DE RECONOCIMIENTO

Ante el Notario Tercero de Barranquilla se presento: WALTER TANAKA TATEKAWA

identificado con CC 16.251.923 PALMIRA

Y declaro que el contenido del anterior Documento es cierto y suya la firma que lo refrenda.

Barranquilla: 14 ABR. 2010

REPÚBLICA DE COLOMBIA

NOTARIA

Rafael J. Pianeta Terraza
NOTARIO ENCARGADO

NOTARIA TERCERA DEL CIRCULO DE B/QUILLA

03-12009291 024802 PROCAPS S.A.
Barranquilla, 14 de Abril de 2010 Hr:14:43:24

No. 14606552

CAMARA DE COMERCIO DE BARRANQUILLA

C A M A R A D E C O M E R C I O
D E B A R R A N Q U I L L A
07**********

CERTIFICADO DE EXISTENCIA Y REPRESENTACION LEGAL O DE INSCRIPCION DE DOCUMENTOS.
PROCAPS S.A.----------
NIT: 890.106.527-5.

EN JUNIO DE ESTE AÑO SE ELEGIRÁ JUNTA DIRECTIVA DE LA CÁMARA DE COMERCIO. LAS INSCRIPCIONES DE CANDIDATOS DEBEN HACERSE EN LA PRIMERA QUINCENA DE MAYO. PARA INFORMACIÓN DETALLADA DIRIGIRSE A LA SEDE PRINCIPAL O COMUNICARSE AL SIGUIENTE TELEFONO: 3303718-3303716.

EL SUSCRITO SECRETARIO DE LA CAMARA DE COMERCIO DE BARRANQUILLA, CON FUNDAMENTO EN LAS INSCRIPCIONES DEL REGISTRO MERCANTIL:

C E R T I F I C A

Que por Escritura Pública No. 1,190 del 22 de Julio de 1976, otorgada en la Notaria Tercera de Barranquilla, inscrito(as) en esta Cámara de Comercio, el 28 de Julio de 1976 bajo el No. 6,070 del libro respectivo, fue constituida la sociedad----------
limitada denominada "PRODUCTORA DE CAPSULAS DE GELATINAS LIMITADA "PROCAPS LIMITADA".----------

C E R T I F I C A

Que por Escritura Pública No. 1,307 del 28 de Junio de 1979, otorgada en la Notaria Tercera de Barranquilla, inscrito(as) en esta Cámara de Comercio, el 23 de Julio de 1979 bajo el No. 10,039 del libro respectivo, la sociedad antes mencionada----------
se transformo en sociedad anonima bajo la denominacion social de "PRODUCTORA DE CAPSULAS DE GELATINA S.A." PROCAPS S.A.".----------

C E R T I F I C A

Que por Escritura Pública No. 3,810 del 31 de Dic/bre de 1980, otorgada en la Notaria Primera de Barranquilla, inscrito(as) en esta Cámara de Comercio, el 30 de Enero de 1981 bajo el No. 12,705 del libro respectivo, la sociedad antes mencionada----------
se transformo en sociedad de responsabilidad limitada bajo al razon social de "PRODUCTORA DE CAPSULAS DE GELATINA LIMITADA "PROCAPS LI - MITADA".----------

C E R T I F I C A

Que por Escritura Pública No. 3,755 del 10 de Nov/bre de 1983, otorgada en la Notaria Unica de Soledad, inscrito(as) en esta Cámara de Comercio, el 22 de Nov/bre de 1983 bajo el No. 17,922 del libro respectivo, la sociedad antes mencionada----------
se transformo en sociedad anonima bajo la denominacion social de "PRODUCTORA DE CAPSULAS DE GELATINA S.A. "PROCAPS S.A.".----------

********** C O N T I N U A **********

CERTIFICADO DE EXISTENCIA Y REPRESENTACION LEGAL O DE INSCRIPCION DE DOCUMENTOS.

PROCAPS S.A.----------
NIT: 890.106.527-5.

C E R T I F I C A

Que por Escritura Pública No. 3,615 del 06 de Dic/bre de 1996, otorgada en la Notaria 2a. de Barranquilla, inscrito(as) en esta Cámara de Comercio, el 20 de Dic/bre de 1996 bajo el No. 67,218 del libro respectivo, la sociedad antes mencionada----------
cambio de razon social, por la denominacion LABORATORIOS PROCAPS S.A
. ----------

C E R T I F I C A

Que por Escritura Pública No. 3,775 del 18 de Dic/bre de 1996, otorgada en la Notaria 2a. de Barranquilla, inscrito(as) en esta Cámara de Comercio, el 23 de Enero de 1997 bajo el No. 67,704 del libro respectivo, la sociedad antes mencionada----------
cambio de razon social, por la denominacion PROCAPS S.A. ----------

C E R T I F I C A

Que dicha sociedad ha sido reformada por las siguientes escrituras y/o documentos privados:

Numero	aaaa/mm/dd	Notaria	No. Insc o Reg	aaaa/mm/dd
39	1979/01/18	Notaria 3a. de Barranquilla.	9,452	1979/01/24
992	1981/04/30	Notaria 1a. de Barranquilla.	13,112	1981/05/06
1,464	1982/08/27	Notaria Unica de Soledad	15,533	1982/09/08
2,744	1983/08/18	Notaria Unica de Soledad	17,416	1983/08/26
3,826	1984/07/16	Notaria Unica de Soledad	19,554	1984/07/27
3,835	1985/08/26	Notaria Unica de Soledad	22,230	1985/09/04
2,260	1986/09/10	Notaria Unica de Soledad	24,995	1986/09/15
1,849	1989/07/11	Notaria 2a. de Barranquilla.	33,909	1989/07/13
444	1990/02/21	Notaria 2a. de Barranquilla.	36,101	1990/03/16
3,090	1991/12/17	Notaria 2a. de Barranquilla	43,993	1992/01/28
269	1993/02/01	Notaria 2a. de Barranquilla	48,428	1993/02/16
2,342	1993/07/28	Notaria 2a. de Barranquilla	50,582	1993/08/10
3,652	1993/11/09	Notaria 2a. de Barranquilla	51,798	1993/11/22
3,615	1996/12/06	Notaria 2a. de Barranquilla	67,218	1996/12/20
89	1997/01/16	Notaria 2a. de Barranquilla	67,704	1997/01/23
2,070	1997/07/17	Notaria 2a. de Barranquilla	70,627	1997/07/31
2,281	1997/08/04	Notaria 2a. de Barranquilla	70,813	1997/08/14
765	1999/04/22	Notaria 3a. de Barranquilla	80,628	1999/04/23
459	2001/02/21	Notaria 2a. de Barranquilla	91,502	2001/02/23
2,608	2001/10/05	Notaria 2a. de Barranquilla	95,771	2001/11/08
1,730	2004/07/29	Notaria 2. de Barranquilla	112,999	2004/08/26
416	2005/03/03	Notaria 2. de Barranquilla	116,364	2005/03/10
1,613	2005/07/28	Notaria 2. de Barranquilla	119,322	2005/08/18
710	2009/05/05	Notaria 3 a. de Barranquilla	150,130	2009/06/30
1,054	2009/06/25	Notaria 3 a. de Barranquilla	150,130	2009/06/30

C E R T I F I C A

Que de acuerdo con la(s) escritura(s) arriba citada(s), la sociedad se rige por las siguientes disposiciones:
DENOMINACION O RAZON SOCIAL:

********** C O N T I N U A **********

03-12009291 024802 PROCAPS S.A.
Barranquilla, 14 de Abril de 2010 Hr:14:43:24

No. 14606553

CAMARA DE COMERCIO DE BARRANQUILLA

C A M A R A D E C O M E R C I O
D E B A R R A N Q U I L L A
07**********

CERTIFICADO DE EXISTENCIA Y REPRESENTACION LEGAL O DE INSCRIPCION DE DOCUMENTOS.
PROCAPS S.A.----------
NIT: 890.106.527-5.

PROCAPS S.A.----------
SIGLA: PROCAPS.
DOMICILIO PRINCIPAL: Barranquilla.
NIT No: 890.106.527-5.
MATRICULA MERCANTIL: 24,802.

C E R T I F I C A

Direccion para notificaciones judiciales:
CL 80 No 78 B - 201.
De la ciudad de Barranquilla.

C E R T I F I C A

DURACION: El término de duración de la sociedad se fijó hasta el 18 de Agosto de 2073.

C E R T I F I C A

OBJETO SOCIAL: El objeto principal de la sociedad sera: a) La fabicacion, importacion y comercializacion de alimentos procesados y suplementos dietarios. b)La fabricacion de capsulas y otros productos similares a partir de materiales tales como gelatinas u otros de distinta naturaleza, que puedan ser utilizados como envases o recipientes de productos y sustancias quimicas, farmaceuticas, alimenticias y cosmeticas, para uso y consumo humano y veterinario. c) La investigacion, desarrollo y fabricacion de toda clase de productos y sustancias quimicas, farmaceuticas, alimenticias y cosmeticas para uso y consumo humano, animal y vegetal. d) La compra y venta de materias primas, insumos y productos intermedios necesarios para los procesos de fabricacion de sustancias quimicas, farmaceuticas, alimen ticias y cosmeticas, asi como la comercializacion interna y externa de los productos terminados. e) La compra, venta, importacion, exportacion y distribucion de toda clase de elementos instrumentales y equipos medicos, quirurgicos, odontologicos y hospitalarios para la salud humana y animal. f) La compra, venta, exportacion, fabricacion, importacion, distribucion de dulces, confites, galletas y todo tipo de golosinas o alimentos para el consumo humano. g) Actuar como representante o agente de personas naturales y juridicas, nacionales y extranjeras en la realizacion de operaciones o transacciones relacionadas con la industria farmaceutica y en especial con cualquiera de las actividades descritas en los literales a), b), c), d) y e) antes descritos. h) Prestar los servicios de asesorias y consultoria en las areas administrativas,

********** C O N T I N U A **********

PROCAPS S.A.----------
NIT: 890.106.527-5.

de mercadeo, analisis clinico, asistencia tecnica, investigacion, costos y produccion, exportaciones, importaciones, ventas, relacionadas con iguales o similares actividades a las que constituyen el objeto de PROCAPS S.A., asi como los de publicidad y promocion de productos quimicos, alimenticios, cosmeticos y farmaceuticos para uso humano, animal o industrial. i) Adquirir, operar, administrar y explotar, a cualquier titulo, empresa o establecimientos dedicados a la investigacion, desarrollo, fabricacion o comercializacion de productos quimicos, farmaceuticos, alimenticios, hospitalarios o cosmeticos. En desarrollo de su objeto principal, la sociedad podra tomar interes social como accionista, socio o fundador de empresas que tengan igual o similar objeto, comprar, vender, importar y exportar maquinarias, insumos y repuestos requeridos para el desarrollo de sus actividades; tomar dinero en prestamo, financiar y garantizar las operaciones comerciales que celebre con terceros relativas a su objeto; celebrar y ejecutar todo tipo de actos y contratos necesarios para desarrollar sus operaciones; girar, aceptar, otorgar y protestar todo tipo de titulos valores e instrumentos negociables, comprar, vender y constituir gravamenes sobre bienes muebles e inmuebles, designar representantes apoderados en el pais y en el exterior y, en general, realizar cualquier acto o negocio que sea necesario o conveniente para el desarrollo de su objeto social y para la mejor proteccion de sus intereses corporativos o la de sus socios. La sociedad podra garantizar el cumplimiento de sus obligaciones adquiridas por terceros o por sus socios, para lo cual podra inclusive constituir gravamenes reales sobre sus bienes de cualquier naturaleza.----------

C E R T I F I C A

CAPITAL	Nro Acciones	Valor Acción
Autorizado		
$**********2,000,000,000	**********2,000,000	**********1,000
Suscrito		
$**********1,229,071,000	**********1,229,071	**********1,000
Pagado		
$**********1,229,071,000	**********1,229,071	**********1,000

C E R T I F I C A

ADMINISTRACION: La direccion y administracion de la sociedad corresponderá a la Asamblea General de Accionistas, a la Junta Directiva, al Presidente, al Vicepresidente y a un Gerente General. Corresponde a la Junta Directiva las siguientes funciones y atribuciones entre otras: Autorizar cualquier acto o contrato cuya cuantia sea superior al equivalente en pesos Colombianos a doscientos cincuenta mil dolares de los Estados Unidos de America (US$250.000), liquidados a la tasa de cambio representativa del mercado vigente el dia en que se surta la transacion. Cuando se trate de operaciones en virtud de las cuales la sociedad garantice obligaciones de terceros o de los socios, se requerira que la operacion sea aprobada con el voto de por lo menos 2/3 partes de los integrantes de la Junta Directiva.

********** C O N T I N U A **********

03-12009291 024802 PROCAPS S.A.
Barranquilla, 14 de Abril de 2010 Hr:14:43:24

No. 14606554

CAMARA DE COMERCIO DE BARRANQUILLA

C A M A R A D E C O M E R C I O
D E B A R R A N Q U I L L A
07**********

CERTIFICADO DE EXISTENCIA Y REPRESENTACION LEGAL O DE INSCRIPCION DE DOCUMENTOS.
PROCAPS S.A.----------
NIT: 890.106.527-5.

La sociedad tendra un Presidente , quien ejercera la representacion legal de la sociedad y tendra las siguientes atribuciones entre otras: Ejecutar las decisiones de la Asamblea General de Accionistas y de la Junta Directiva. Ejercer la Representacion Legal de la sociedad en todos los actos y contratos que se requieran para el desarrollo del objeto social, de conformidad con lo previsto en las leyes y en los presentes estatutos. Constituir apoderados judiciales y extrajudiciales, recibir notificaciones, absolver interrogatorios de parte, confesar obligaciones de la sociedad, transigir, disistir y allanar a la sociedad en cualquier asunto judicial. Aprobar y celebrar en nombre de la socieda todo acto o contrato cuya cuantia sea igual o inferior al equivalente en pesos colombianos a doscientos cincuenta mil dolares de los Estados Unidos de America (US$250.000), liquidados a la tasa de cambio representativa del mercado vigente el dia en que se surta la transacion o celebracion del acto. Las demas funciones que le correspondan como Representante Legal por disposicion de la ley, de estos estatutos o de la Junta Directiva. El Presidente tendra un suplente, designado por la Junta Directiva, quien reemplazara al Presidente en sus faltas temporales, absolutas o meramente accidentales, con entidades facultades y sin que sea necesario para ello acreditar la ausencia del principal. La sociedad tendra un Vicepresidente designado por la Junta Directiva, quien tambien ejercera la representacion legal de la sociedad, pudiendo ejercerla conjunta o separadamente con el Presidente. El vicepresidente tendra las siguentes facultades entre otras. Ejecutar dentro de su competencia las decisiones de la Asamblea General de Accionistas, de la Junta Directiva y del Presidente. Ejercer la representacion legal de la sociedad en todos los actos y contratos que se requieran para el desarrollo del objeto social, de conformidad con lo previsto en las leyes y en los presentes estatutos. Constituir apoderados judiciales y extrajudiciales, recibir notificaciones, absolver interrogatorios de parte, confesar obligaciones de la sociedad, transigir, desistir y allanar a la sociedad en cualquier asunto judicial. Aprobar y celebrar en nombre de la sociedad todo acto o contrato cuya cuantia sea igual o inferior al equivalente en pesos colombianos a ciento cincuenta mil dolares de los Estados Unidos de America (US$150.000), liquidados a la tasa de cambio representativa del mercado vigente el dia en que se surta la aprobacion o celebracion del acto, siempre que no se trate de enajenar bienes inmuebles o cualquier otro activo fijo de la sociedad, inclusive de naturaleza muebles, acciones, cuotas o partes de interes inversiones y todo tipo de derechos de la sociedad, o de constituir gravamenes sobre cualquiera de los anteriores, facultades que radican exclusiva y privativamente en cabeza del Presidente. Las demas funciones que le corresponda como

********** C O N T I N U A **********

CERTIFICADO DE EXISTENCIA Y REPRESENTACION LEGAL O DE INSCRIPCION DE DOCUMENTOS

PROCAPS S.A.----------
NIT: 890.106.527-5.

Representante legal por disposicion de la ley, de estos estatutos o de la Junta Directiva. El Vicepresidente tendra (1) suplente, designado por la Junra Directiva, quien lo reemplazara conjunta o separadamente en sus faltas temporales, absolutas o meramente accidentales, con identicas facultades y sin que sea necesario para ello acreditar la ausencia del principal. La sociedad tendra un (1) Gerente General, con su respectivo Suplente, designados por la Junta Directiva. El Suplente reemplazara al Gerente General, en sus faltas temporales, absolutas o meramente accidentales, quien junto con el gerente general, llevaran la representacion legal de la sociedad, con identicas facultades, pudiendolas ejercer conjunta o separadamente con el Presidente y Vicepresidente, con identicas facultades que el Gerente General y sin que sea necesario para ello acreditar la ausencia del titular. El Gerente General tendra las siguientes facultades entre otras: Ejercer la representacion legal de la sociedad, en todos los actos y contratos que se requieran para el desarrollo del objeto social, de conformidad con lo previsto en las leyes y en los presentes estatutos. Aprobar y celebrar en nombre de la sociedad todo acto o contrato cuya cuantia sea igual o inferior al equivalente en pesos Colombianos a Cincuenta Mil dolares de los Estados Unidos de America (US$50.000), liquidados a la tasa de cambio representativa del mercado vigente el dia en que se surta la aprobacion o celebracion del acto, siempre que no se trate de enajenar a cualquier titulo bienes inmuebles o cualquier otro activo fijo de la sociedad, inclusive de naturaleza mueble, acciones, cuotas o partes de interes, inversiones y todo tipo de derechos de la sociedad o de constituir gravamenes sobre cualquiera de las anteriores facultades que radican exclusiva y privativamente en cabeza del Presidente. ----------

C E R T I F I C A

Que según Acta No. 116 del 27 de Junio de 1997 correspondiente a la Asamblea de Accionistas en Barranquilla, de la sociedad:
PROCAPS S.A.----------
cuya parte pertinente se inscribió en esta Cámara de Comercio, el 15 de Agosto de 1997 bajo el No. 70,838 del libro respectivo, y según Acta No. 127 del 22 de Marzo de 2000 correspondiente a la Asamblea de Accionistas en Barranquilla, de la sociedad:
PROCAPS S.A.----------
cuya parte pertinente se inscribió en esta Cámara de Comercio, el 29 de Mayo de 2000 bajo el No. 87,250 del libro respectivo, fueron hechos los siguientes nombramientos:

CLASE: JUNTA DIRECTIVA

Principales

1. Minski Gontovnik Ruben	CC.*******7,463,982
2. Minski Gontovnik Meyer	CC.*******7,461,324
3. Minski Gontovnik Jose	CC.*******8,671,834

Suplentes

1. Minski Zirberblum Elliot	CC.******72,219,541

********** C O N T I N U A **********

03-12009291 024802 PROCAPS S.A.
Barranquilla, 14 de Abril de 2010 Hr:14:43:24

No. 14606555

CAMARA DE COMERCIO DE BARRANQUILLA

C A M A R A D E C O M E R C I O
D E B A R R A N Q U I L L A
07**********

CERTIFICADO DE EXISTENCIA Y REPRESENTACION LEGAL O DE INSCRIPCION DE DOCUMENTOS.
PROCAPS S.A.----------
NIT: 890.106.527-5.

2. Minski Deitel Daniel .**********
3. Minski Sharon PA.*990,212,082,805

C E R T I F I C A

Que según Acta No. 116 del 27 de Junio de 1997 correspondiente a la Asamblea de Accionistas en Barranquilla, de la sociedad: PROCAPS S.A. cuya parte pertinente se inscribió en esta Cámara de Comercio, el 15 de Agosto de 1997 bajo el No. 70,838 del libro respectivo, fueron hechos los siguientes nombramientos:

Cargo/Nombre	Identificación
Presidente.	
Minski Gontovnik Ruben	CC.*******7,463,982
Suplente del Presidente	
Minski Minski Abraham	CC.*********802,575
Vicepresidente	
Minski Gontovnik Meyer	CC.*******7,461,324
Suplente del vicepresidente	
Minski Gontovnik Jose	CC.*******8,671,834

C E R T I F I C A

Que según Acta No. 11 del 01 de Febrero de 2001 correspondiente a la Junta Directiva en Barranquilla, de la sociedad: PROCAPS S.A. cuya parte pertinente se inscribió en esta Cámara de Comercio, el 12 de Febrero de 2001 bajo el No. 91,273 del libro respectivo, fueron hechos los siguientes nombramientos:

Cargo/Nombre	Identificación
Gerente General.	
Uribe Ochoa Guillermo Luis	CC.******70,073,754

C E R T I F I C A

Que según Acta No. 406 del 02 de Sep/bre de 2004 correspondiente a la Junta Directiva en Barranquilla, de la sociedad: PROCAPS S.A. cuya parte pertinente se inscribió en esta Cámara de Comercio, el 09 de Sep/bre de 2004 bajo el No. 113,263 del libro respectivo, fueron hechos los siguientes nombramientos:

Cargo/Nombre	Identificación
Suplente del Gerente.	
Tanaka Tatekawa Walter	CC.******16,251,923

C E R T I F I C A

Que según Acta No. 124 del 29 de Marzo de 1999 correspondiente a

********** C O N T I N U A **********

CERTIFICADO DE EXISTENCIA Y REPRESENTACION LEGAL O DE INSCRIPCION DE DOCUMENTOS.

PROCAPS S.A.----------
NIT: 890.106.527-5.

la Asamblea de Accionistas en Barranquilla, de la sociedad: PROCAPS S.A. cuya parte pertinente se inscribió en esta Cámara de Comercio, el 04 de Agosto de 1999 bajo el No. 82,226 del libro respectivo, fueron hechos los siguientes nombramientos:

Cargo/Nombre	Identificación
Revisor Fiscal Ppal.	
Vargas Pertuz Ana Isabel	CC.******32,696,645

C E R T I F I C A

Que según Acta No. 151 del 31 de Marzo de 2009 correspondiente a la Asamblea de Accionistas en Barranquilla, de la sociedad: PROCAPS S.A. cuya parte pertinente se inscribió en esta Cámara de Comercio, el 30 de Julio de 2009 bajo el No. 151,129 del libro respectivo, fueron hechos los siguientes nombramientos:

Cargo/Nombre	Identificación
Suplente del Revisor Fiscal.	
Duarte Diaz David	CC.******73,115,965

C E R T I F I C A

Que por Escritura Pública No. 2,784 del 07 de Dic/bre de 2007, otorgada en la Notaria 2 a. de Barranquilla cuya parte pertinente se inscribió en esta Cámara de Comercio, el 20 de Dic/bre de 2007 bajo el No. 3,492 del libro respectivo,---------- y las inscripciones 3.492-1, 3.493, 3.494, 3.495, 3.496, 3.497, ---- 3.498, 3.499, 3.500, 3.501, 3.502, 3.503, 3.504, 3.505, 3.506, ----- 3.507, Consta que RUBEN MINSKI GONTOVNIK, C.C. No. 7.463.982, ------ obrando como Presidente y Representante Legal de la sociedad C.I. NATURMEGA S.A., que obrando en su condicion de Presidente y ------- Representante Legaql de la sociedad PROCAPS S.A., el presidente ---- debidamente facultado por los Estatutos Sociales y por el Maximo Organo Social, otorga los siguientes poderes: Otorgar poder -------- general, amplio y suficiente a los doctores JOSEFINA DEL CARMEN ---- MIELES BUELVAS CC.No.33.138.497; a MARCELA CARVAJALINO PAGANO ------ CC.No.22.579.748; CRISTINA VIOLI FRANCESCHINI CC.No.22.463.716, con las facultades que se transcriben a continuacion: a.- Para que ----- representen a la sociedad PROCAPS S.A, ante todo tipo de ---------- autoridades administrativas y jurisdiccionales, en asuntos de ------ naturaleza civil, laboral, comercial, penal, aduanera, cambiaria, tributaria, policiva, administrativa y fiscal. b.- Contestar y ----- presentar demandas, requerimientos, recursos, pedir y aportar ------ pruebas, recibir, notificarse, ejercer la accion de tutela, -------- tramitar incidente de desacato, comparecer en los procesos que ----- cursen contra la sociedad, allanarse en nombre de la sociedad en los asuntos que sean necesarios, transigir, desistir, sustituir, renunciar y reasumir el poder. c.- La facultad expresa de ---------- conciliar, por tanto pueden llegar a acuerdos conciliatorios, ------ transaccionales, judiciales o extrajudiciales o extrajudiciales, cualquiera que sea su cuantia, para lo cual las apoderadas deben aportar la autorizacion simple y escrita del Representante legal de la sociedad. d.- Podran presentar solicitudes de devolucion y/o compensacion de impuestos, ventas y rentas, recibir los pagos por

********** C O N T I N U A **********

CERTIFICADO DE EXISTENCIA Y REPRESENTACION LEGAL O DE INSCRIPCION DE DOCUMENTOS.
PROCAPS S.A.----------
NIT: 890.106.527-5.

las devoluciones y adelantar todo tramite o proceso administrativo ante la DIAN, de tal forma que en ningun momento quede sin --------- representacion legal de la sociedad. PODERES ESPECIALES: a.- Se ---- confiere poder especial amplio y suficiente a las personas que se determinan seguidamente, que conforman el grupo A y B, para, para que conjuntamente suscriban contratos de suministros de mercancias y servicios con entidades oficiales, hasta la suma de OCHOCIENTOS MIL DOLARES DE LOS ESTADOS UNIDOS DE AMERICA (US800.000.00), ------- liquidados a la tasa representativa del mercado vigente en el dia que se realice la respectiva transaccion. Grupo A. WALTER NANAKA TATEKAWA CC.No.16.251.923, CLAUDIA TANGARIFE CASTILLO ---------- CC.No.41.660.293, CARMEN ALICIA FLOREZ CC.No.22.434.044, IVAN ------ VILLAREAL CAMACHO CC.No.91.201.749, CARMEN ALICIA HERRERA ---------- DIAZGRANADOS CC.No.22.377.540, DAVID ANTONIO CHARRIS GIRALDO ------- CC.No.19.341.274, RICARDO DORADO HURTADO CC.No.79.715.772, JAIME FORERO LLINAS CC.No.19.300.593. b.- Se confiere poder especial ----- amplio y suficiente al senor WILLIAM ALBERTO BARROS CERRA ---------- CC.No.8.715.941, para que en nombre y representacion de PROCAPS ---- S.A., suscriban declaraciones cambiarias, aduaneras y tributarias a que este obligada la sociedad, en el giro normal de sus negocios y operaciones sociales, por tanto quedan facultados para firmarlas, como tambien los demas documentos relacionados con ellas, ---------- notificarse, aportar y solicitar las pruebas que sean necesarias. c.-Otorgar poder especial amplio y suficiente al senor MARLON ----- TATIS PAREDES CC.No.72.176.874 y al senor WILLIAM ALBERTO BARROS CERRA CC.No.8.715.941, para que en nombre y representacion de ------ PROCANPS S.A., suscriban las declaraciones, reportes y cualquier documento relacionado con los tramites o registros de Comercio ----- Exterior y o cualquier entidad del Estado que haga sus veces: d) Otorgar poder especial amplio y suficiente a la doctora CARMEN ----- ALICIA HERRERA DIAZGRANADOS, CC.No.22.377.540 y al doctor WALTER TANAKA TATEKAWA, CC.No.16.251.923, para que en nombre y ---------- representacion de PROCAPS S.A.; endosen los titulos valores de la sociedad hasta por la suma de UN MIL MILLONES DE PESOS ---------- (1.000.000.000.00) M.L. CUARTO: EI representante legal de la ------- sociedad PROCAPS S.A.; doctor RUBEN MINSKI GONTOVNIK, manifiesta que la presente reforma fue aprobada por la Asamblea General ------- Extraordinaria de Accionista de PROCAPS S.A., con observancia de las disposiciones legales y estatutarias.----------

C E R T I F I C A

Que por Escritura Pública No. 1,933 del 16 de Sep/bre de 2008, otorgada en la Notaria 2 a. de Barranquilla cuya parte pertinente se inscribió en esta Cámara de Comercio, el 06 de Octubre de 2008

********** C O N T I N U A **********

CERTIFICADO DE EXISTENCIA Y REPRESENTACION LEGAL O DE INSCRIPCION DE DOCUMENTOS.

PROCAPS S.A.----------
NIT: 890.106.527-5.

bajo el No. 3,748 del libro respectivo,----------
Consta que el señor RUBEN MINSKI GONTOVNIK, C.C. No. 7.463.982, que comparece en su condicion de Presidente y Representante Legal de la Sociedad PROCAPS S.A., confiere por medio de este instrumento ------ publico Poder especial Amplio y Suficiente a la doctora MILENA ----- ESCAFFI PARDO, C.C. No. 32.758.420, para que en nombre y ---------- representacion de PROCAPS S.A., suscriba declaraciones cambiarias, aduaneras y tributarias a que este obligada la sociedad en el giro normal de sus negocios y operaciones sociales, por tanto queda ----- facultada para firmar, como tambien los demas documentos ---------- relacionados con ellas, notificarse, aportar y solicitar las ------- pruebas que sean necesarias.----------

C E R T I F I C A

Que en esta Cámara de Comercio no aparecen inscripciones posteriores de documentos referentes a reforma, disolución, liquidación o nombramientos de representantes legales de la expresada sociedad.

C E R T I F I C A

Que su última Renovación fue el: 30 de Marzo de 2010.

La información sobre embargos de establecimiento se suministra en Certificados de Matrícula, la de contratos sujetos a registro, en Certificados Especiales.

C E R T I F I C A

Que los actos de registro aquí certificados quedan en firme cinco días hábiles despues de la correspondiente inscripción, siempre que, dentro de dicho término, no sean objeto de los recursos de reposición ante esta entidad, y/o de apelación para ante la Superintendencia de Industria y Comercio.

US006723358B1

(12) **United States Patent**
van Lengerich

(10) **Patent No.: US 6,723,358 B1**
(45) **Date of Patent: Apr. 20, 2004**

(54) **ENCAPSULATION OF COMPONENTS INTO EDIBLE PRODUCTS**

(75) Inventor: **Bernhard H. van Lengerich**, Plymouth, MN (US)

(73) Assignee: **General Mills, Inc.**, Minneapolis, MN (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: **09/673,983**

(22) PCT Filed: **Mar. 23, 1999**

(86) PCT No.: **PCT/US99/04267**

§ 371 (c)(1),
(2), (4) Date: **Feb. 1, 2001**

(87) PCT Pub. No.: **WO99/48372**

PCT Pub. Date: **Sep. 30, 1999**

Related U.S. Application Data

(63) Continuation-in-part of application No. 09/233,443, filed on Jan. 20, 1999.

(60) Provisional application No. 60/109,696, filed on Nov. 24, 1998, provisional application No. 60/103,700, filed on Oct. 9, 1998, and provisional application No. 60/079,060, filed on Mar. 23, 1998.

(51) **Int. Cl.**[7] **A21D 13/00**

(52) **U.S. Cl.** **426/94**; 426/61; 426/89; 426/549; 424/439

(58) **Field of Search** 426/94, 61, 89, 426/549, 96, 285, 302; 424/439

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(57) **ABSTRACT**

An edible matrix composition that has a chewable texture and that contains at least one encapsulated component is obtained by admixing at least one plasticizer, and a ground, free-flowing particulate mixture which comprises at least one fat, at least one starch, and at least one sugar which have been mixed and heated without substanitially gelatinizing the starch. A chewable texture is obtained rather than a hard, glassy matrix because the starch is substantially ungelatinized. However, a flavorful product is obtained without destroying a heat sensitive encapsulant because the starch is admixed with ingredients comprising fat or oil and sugar and the mixture is heated to develop flavor at high temperatures prior to admixing with the heat sensitive encapsulant. The encapsulated component may be at least one biologically active component, pharmaceutical component, nutraceutical component, or microorganism. In preferred embodiments, the free-flowing mixture is obtained by grinding cookies. The free-flowing mixture, such as ground cookies and the plasticizer, such as oil and water are mixed with an encapsulant to obtain a formable dough or crumbly mass. The formable dough is shaped or formed into pieces or pellets and dried to a shelf-stable moisture content

67 Claims, No Drawings

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ENCAPSULATION OF COMPONENTS INTO EDIBLE PRODUCTS

This application is a 371 application of PCT/US99/04267 filed Mar. 23, 1999 and is a continuation in part of application Ser. No. 09/233,443 filed Jan. 20, 1999, and claims the benefit of provisional application Nos. 60/079,060 filed Mar. 23, 1998, No. 60/103,700 filed Oct. 9, 1998 and No. 60/109,696 filed Nov. 24, 1998.

FIELD OF THE INVENTION

The present invention relates to compositions, the use and the manufacturing of edible products, that contain encapsulated or embedded components, such as nutraceutical components, pharmaceuticals, biologically active components, and/or live microorganisms. The products according to this invention are intended to be consumed either directly as a food or they may be used as additions to food, such as topical applications or in beverages. They are not intended to undergo additional severe processing that might thermally or mechanically destroy the encapsulant. The products made according to this invention exhibit eating qualities, such as a chewable texture, as it occurs in for example streusel or chewable vitamin pills, and a cookie-like taste.

BACKGROUND OF THE INVENTION

Encapsulation of food components is described in *Encapsulation and Controlled Release of Food Ingredients*, edited by S. J. Risch and G. A. Reineccius, ACS Symposium Series 590 (1995). U.S. Pat. No. 5,183,690 to Carr, et. al. describes a continuous extrusion process using starch-based material to encapsulate components. The resulting products are in the form of particulates, which have gelatinized starch as a continuous domain, in which discontinuous domains of biologically active core material is entrapped.

Encapsulation of heat sensitive components, for example nutraceutical components, such as for example microorganisms, into matrixes that are edible, is generally difficult for a number of reasons. First, conventional encapsulation processes which expose matrix material and encapsulants to high temperatures, causes thermal destruction or loss of encapsulant. Thus, either large overdoses of encapsulant, which would be very expensive, would be required, or the encapsulant would not sustain the encapsulation process at all. Second, if the encapsulant can be encapsulated into a matrix under sufficiently low temperatures, the resulting product is a solid, that is characterized as substantially hard and glass-like. The hardness and glassiness is caused by cooking a starch-based material with a sufficient amount of water to gelatinize the starch and subsequently separating the starch-based materials into discrete particles and drying them so that the water content of the starchy mass is sufficiently low. However, the temperature at which the particles are consumed, or the eating temperature, is generally lower than 50° C., which is far below the glass transition temperature, T_g, of the cooked, dried starch. Therefore, products of this kind exhibit a dense and glassy, very hard texture, that may be very suitable to encapsulate components. However, when chewed they cause severe problems, because they are not chewable and exhibit a texture similar to that of uncooked rice or pasta. They can therefore be only swallowed as microtablets without chewing. They could also be used as dense pellets for a variety of processing applications, where a controlled release of the heat sensitive encapsulant is desired. The physical hardness

of the products and their mechanical stability are advantageous for many processing applications. However, chewing or masticating of these products would be very unpleasant and their incorporation into other food products is not practical.

The present invention provides an edible product, that is chewable, has a pleasant taste and texture and contains encapsulated components, particularly nutraceutical, pharmaceutical or biologically active components. The chewable product can be used either as a food product itself, or as part of a food product, i.e. as an ingredient or as a topping, that may be applied to surfaces of food. A main physical characteristic of the product made according to the present invention is that the product is substantially less hard than foods products such as uncooked rice and raw pasta. The chewable, flavorful product can be eaten either alone as a food itself, or for example as medical food, as food having a pharmaceutical effect, as a dietary supplement, or in combination with other foods, such as a topping or in pasty foods or beverages.

SUMMARY OF THE INVENTION

The present invention provides a product that contains an encapsulated nutraceutical component, pharmaceutical component, biologically active component, or live microorganisms, or a combination thereof. In preferred embodiments, the product contains an encapsulated microorganism. The products of the present invention contain the one or more encapsulants in a pleasantly tasting and chewable surrounding matrix.

The matrix composition of the present invention comprises a plasticizer, such as oil and/or water, and a substantial amount of a free flowing mixture of at least one fat, one starch and one sugar, that are preprocessed, i.e. mixed and heat treated so as to substantially prevent gelatinization of starch and to provide a pleasantly tasting product. An example of the free flowing mixture is fine ground-up cookies having a particle size of 100% smaller than about 1 mm.

The mixture may contain additional components to enhance preprocessing or to improve sensory attributes, such as flavor, sodium chloride, nonfat dry milk, whey protein, high fructose corn syrup, dextrins, and leavening agents, as well as other components known to those skilled in the art of producing pleasantly tasting cookie type products.

The matrix may further comprise components that either facilitate processing, or mask the unpleasant taste of the encapsulants, or prevent exposure to oxygen or air, or which enhance the sensory attributes of the final product. These components include, for example, lipids, such as oils or fats, chocolate liquor, chocolate, cocoa powder, compound coatings, flavors, concentrated fruit juice, or particulates, such, as for example ground nuts or almonds. The water may be pH adjusted to obtain a good tasting product.

A chewable texture is obtained rather than a hard, glassy matrix because the starch is substantially ungelatinized. However, a flavorful product is obtained without destroying a heat sensitive encapsulant because the starch is admixed with ingredients comprising fat or oil and sugar and the mixture is heated to develop flavor at high temperatures prior to admixing with the heat sensitive encapsulant.

In accordance with the method of the present invention, a product containing a substantial amount of a free-flowing material in which the starch component is substantially ungelatinized may be obtained by grinding a baked product,

such as cookies or cookie-type products, to obtain a free-flowing particulate mixture or flour. The reduced particle size facilitates the formation of a dough or crumbly mass upon mixing of the free-flowing mixture with a plasticizer such as water and/or oil. In embodiments of the invention, the particle size of a cookie-type product or cookies may be reduced to obtain a dry, free-flowing particulate mixture having a particle size of about 100% smaller that 1 mm.

Admixing of the free-flowing mixture, such as ground-up cookie flour, with at least one plasticizer such as water and/or oil and the encapsulant with the other components to obtain a dough or crumbly mass may be performed continuously using an extruder or continuous mixer. The dough or crumbly mass may be formed or pressed into discrete particles. Moisture may be removed from the particles to an amount sufficiently low so as to obtain a sufficient shelf life. The products made according to the invention exhibit a granular, crumbly structure having a pleasant taste and are chewable.

DETAILED DESCRIPTION OF THE INVENTION

An edible, starch-based matrix composition that contains at least one encapsulated component and that is chewable, rather than hard and glassy is obtained from a ground pre-processed, free-flowing particulate mixture and at least one plasticizer without substantially gelatinizing the starch. The free-flowing mixture may be obtained by admixing and heating at least one fat, at least one starch, and at least one sugar without substantially gelatinizing the starch. The encapsulated component may be one or more biologically active components, pharmaceutical components, nutraceutical components, microorganisms, or mixtures thereof. In embodiments of the invention, the free-flowing mixture may be obtained by baking a mixture of said at least one fat, at least one starch, and at least one sugar without substantially gelatinizing said at least one starch to obtain a baked product and then grinding the baked product to obtain the free-flowing mixture. In preferred embodiments, the free-flowing mixture is at least substantially ground cookies or a flour obtained by grinding cookies.

A key requirement of the free-flowing mixture, such as ground-up cookie flour or ground-up cracker flour is that its starch component, which may originate from grain sources such as wheat, oats, barley, corn, rye, or other grains or potatoes or other roots, is substantially not gelatinized. Production of a free-flowing mixture without substantially gelatinizing starch can be accomplished in known manner by any one ordinarily skilled in the art of making cookies or cookie-type products. If the free-flowing mixture contained substantial amounts of gelatinized starches, the process would result in products that after drying exhibit substantial glassiness, hardness and thus a mechanical stability that is undesirable. The substantially gelatinized, glassy, hard products would not exhibit desirable sensory attributes.

If the matrix composition was made from a pasta flour, such as for example semolina, the mechanical characteristics of the final product would be similar to commonly known uncooked pasta and would be undesirably hard for direct consumption. Products made from soft wheat flour that has not been cooked or processed into a cookie-type product, would exhibit inferior taste and would be undesirable for direct consumption.

A cookie-type product for use in the present invention may be made by known conventional cookie production processes. As is well known, cookie production comprises admixing of at least fat and sugar, and adding flour to the premixed sugar/fat mix to obtain a cookie dough. Optionally, minor ingredients and water can be added to facilitate machining of the dough, and to obtain a typical texture and taste of the final cookie. The cookie dough is formed into individual pieces and baked under conventional baking conditions. The baking is generally performed above the gelatinization temperature of starch, but the starch is not substantially gelatinized because sufficient amounts of water are not accessible to the starch at the elevated temperatures. However, the baking helps to develop flavor, through Maillard reactions for example, and develops a leavened, crumb structure.

Conventional or commercially available cookie or other baked good formulations and ingredients may be used to produce the free-flowing mixtures employed in the present invention. Examples of cookies and cookie-like products which may be ground for use in the present invention are butter cookies, sugar cookies, graham crackers, chocolate chip cookies, oatmeal cookies, sugar wafers, almond cookies, chocolate cookies, vanilla wafers, mixtures thereof, and the like.

Exemplary amounts of ingredients which may be used to obtain a free-flowing mixture are: from about 10% by weight to about 40% by weight fat, from about 20% by weight to about 40% by weight sugar, and from about 45% by weight to about 75% by weight flour, preferably wheat flour, based upon the total weight of the fat, sugar and flour in the free-flowing mixture.

Conventional minor and other conventional cookie ingredients, which may be included in the free-flowing mixture, such as ground cookies, are for example, high fructose corn syrup, maltodextrins, corn syrup, dextrose, maltose, modified or unmodified starches, eggs, leavening agents such as sodium bicarbonate, ammonium bicarbonate baking soda, and calcium phosphate, non-fat dry milk, full-fat dry milk, whey proteins, gluten, natural or artificial flavors, insoluble and soluble fibers, such as inulin, hydrocolloids, such as guar gum or gum arabic, dry eggs, salt, nutrients, and emulsifiers such as mono- and diglycerides.

In embodiments of the invention, ground cookies employed as a free-flowing mixture in the present invention may comprise from about 8% by weight to about 40% by weight shortening or fat, from about 15% by weight to about 40% by weight sugar, and from about 20% by weight to about 75% by weight flour, based upon the weight of the ground cookies.

In embodiments of the invention, after baking and cooling, a cookie product or a commercially available cookie may be ground up into a flour or powder or meal using a conventional mill or other grinding apparatus. The particle size distribution may preferably be similar to that of a flour. The ground flour has a preferred particle size of 100% smaller than 1 mm to form a continuous dough phase. In embodiments of the invention, the ground, particulate free-flowing mixture may have a particle size distribution of 100% less than 10 mm. However, with a much coarser particle size, (for example if the majority of the particles were 5 mm) the ground product would not yield a uniform dough and would produce a granular, nonhomogeneous phase. Much finer powders, for example 100% less than 50 micron would create processing difficulties, such as feeding problems, dusting, and caking to processing surfaces. In preferred embodiments at least a substantial portion of the ground, free-flowing particulate mixture has a particle size distribution of from about 50 microns to about 1 mm.

Another requirement of the free-flowing mixture such as ground cookies is that its starch component is substantially ungelatinized. This may be accomplished, during the process of cookie making for example, by mixing and/or coating the starch-containing components with fat before adding optional moisture. Another possibility is to entrap added moisture in fat and prevent moisture access to the starch in the flour. At least substantially ungelatinized starch in the final ground cookie or other free-flowing mixture tends to resist subsequent gelatinization when mixed with water. In embodiments of the invention the starch in the free-flowing mixture, such as ground cookies, and in the matrix compositions of the present invention has a degree of starch gelatinization of less than about 50%, preferably less than about 30%, preferably less than about 15%, as measured by differential scanning calorimetry (DSC).

The free-flowing mixture, such as ground cookies is used in an effective encapsulating amount. In embodiments of the present invention, the free-flowing mixture content, such as the cookie or cracker flour content of the particles may be at least about 10% by weight, generally at least about 30% by weight, for example from about 60% by weight to about 95% by weight, based upon the weight of the edible, chewable matrix composition. The amount of free-flowing mixture, such as ground cookies employed may depend upon the desired texture and the plasticizer employed. For example, if water is employed as the plasticizer, generally the free-flowing mixture content of the chewable matrix composition is higher, for example, at least about 40% by weight, because water is removed during drying to obtain a shelf stable product. If the plasticizer employed does not have to be removed to obtain a shelf-stable product, the free-flowing mixture content may be lower. For example, if the plasticizer is a fat which is at least substantially solid at room temperature, the free-flowing mixture content may be at least about 10% by weight, based upon the weight of the edible, chewable matrix composition.

Plasticizers employed in the present invention may be any edible or consumable liquid which enables the formation of a substantially homogeneous cohesive, plasticized, viscoelastic, formable mixture, dough or crumbly mass. Exemplary of plasticizers which may be used are water, an aqueous-based composition such as a sugar solution, juice, alcohol, glycerol, and sorbitol, oils, melted shortenings or fat, and mixtures thereof.

The amount of liquid plasticizer, such as water and/or oil, should generally be sufficient to obtain a formable mixture or dough at a sufficiently low temperature and under sufficiently low shear conditions so as to avoid substantial mechanical or thermal destruction of the free-flowing mixture or encapsulant. Exemplary total amounts of plasticizer, such as oil and/or water, used to form a dough or crumbly mass may range up to about 90% by weight, generally from about 10% by weight to about 70% by weight, for example from about 20% by weight to about 45% by weight, based upon the total weight of the free-flowing mixture, such as ground cookies, and the added plasticizer used to form the dough or crumbly mass.

If water is employed as a plasticizer, it may generally be used in an amount of less than or equal to about 25% by weight, based upon the total weight of the free-flowing mixture and added water to obtain a formable, extrudable dough. Higher amounts are less desirable, because more drying may be needed to obtain a shelf-stable product. When an edible oil, shortening or fat is employed as a plasticizer, it may generally be used in an amount of up to about 90% by weight, preferably up to about 20% by weight, most preferably up to about 10% by weight, based upon the total weight of the free-flowing mixture, such as ground cookies and the oil, shortening or fat.

Generally, higher amounts of the encapsulant component may be encapsulated in the matrix composition when an oil, shortening or fat is used as a plasticizer because generally higher amounts of oil may be employed compared to the amount of water which may be employed. Use of an oil or fat as a plasticizer in place of or in addition to water is advantageous because the oil or fat in addition to facilitating extrusion, also serves as a preencapsulation medium. The oil or fat provides a protective coating on the free-flowing mixture, such as ground cookies, and on the encapsulant. Also, the need for drying of the dough to obtain a shelf-stable moisture content is substantially reduced or eliminated when oil or fat is employed as a plasticizer. The addition of vegetable oil during mixing has been proven useful to obtain a smooth continuous dough phase and it facilitates forming of the dough into discrete particles.

Edible oils, shortenings or and fats which may be employed include those derived from plant, animal, and marine sources such as vegetable shortening or oils, which include corn oil, safflower oil, soybean oil, and cotton seed oil, which may be hydrogenated, as well as edible fat substitutes. In embodiments of the invention, particularly where high levels of oil or fat are employed, the melting point of the oil, shortening or fat should be sufficiently high so as to avoid separation of oil during extrusion. For example, the melting point of the oil, shortening or fat may be at least about 30° C., preferably at least about 37° C., most preferably at least about 40° C.

In embodiments of the invention, the formable mixture or dough may have a total plasticizer content, such as water and/or oil, of up to about 90% by weight, generally from about 10% by weight to about 50% by weight, for example about 15% by weight to about 25% by weight. The total plasticizer content may include water supplied by any liquid encapsulant component and additional plasticizer, such as added water, glycerol, sorbitol or a combination thereof or any other liquids, such as fruit juice, that enables the formation of a dough. When water or low melting point oils are employed at high levels, for example a moisture content well above 50%, a thin, low viscosity dough may result. The low viscosity dough may either not be formable or the drying efforts would be unnecessarily high. Substantially lower moisture contents, such as well below 5% may result in a dry product, which would be too fragile after forming and would fall apart. It may also generate frictional heat during extrusion forming which would be detrimental to the heat sensitive encapsulant. The water may be mixed with organic acids or fruit juice to adjust pH and to obtain a pleasant flavor in the final product.

In embodiments of the invention, where for example, removal of plasticizer is not needed to obtain a shelf-stable matrix composition, the plasticizer content of the edible, chewable matrix composition may be at least substantially the same as the plasticizer content of the formable mixture, dough or crumbly mass. For example, in embodiments of the invention when an oil, shortening or fat is employed as a plasticizer, the plasticizer content of the edible, chewable matrix composition may be up to about 90% by weight, for example from about 10% by weight to about 50% by weight, based upon the weight of the matrix composition.

The liquid plasticizer content of any liquid encapsulant component utilized may be at least about 35% by weight, generally at least about 50% by weight, for example from

about 65% by weight to about 90% by weight, based upon the weight of the liquid encapsulant component.

For example, an aqueous dispersion of *Lactobacillus acidophilus* may have a moisture content of about 70% by weight and an encapsulant content (*Lactobacillus acidophilus*) of about 30% by weight. The 70% moisture content stemming from the acidophilus dispersion may be used as a plasticizer. The ratio of the free-flowing mixture to moisture stemming from the aqueous encapsulant liquid may be about 3:1 to enable the formation of a homogeneous dough. Vegetable oil may be added to delay penetration of water into the matrix and delay the release of the microorganism. The encapsulation of sensitive liquid components into a matrix to obtain discrete shelf-stable particles is disclosed in U.S. patent application Ser. No. 09/233,443 filed Jan. 20, 1999 in the name of Bernhard H. van Lengerich for "Encapsulation of Sensitive Liquid Components into a Matrix to Obtain Discrete Shelf-stable Particles," the disclosure of which is herein incorporated by reference in its entirety.

Edible, plasticizable matrix components which form a glassy matrix, such as gelatinized starches, and other ingredients may be included in the chewable, matrix compositions of the present invention provided they do not adversely affect the chewable texture of the matrix composition. Gelatinized starches may, for example, be included in an amount up to about 30% by weight of the free-flowing mixture, such as ground cookies.

Examples of optional, edible, plasticizable matrix materials which are plasticizable at low temperatures by the plasticizer component may be a plasticizable biopolymer such as a carbohydrate, such as a starch or cyclodextrin, polyethylene glycol, pentosans, hydrocolloids such as carragenan, alginates, or gum arabic, wheat gluten, such as vital wheat gluten or isolated gluten, and mixtures thereof. Exemplary starches which may be used are native or modified starches or pregelatinized starches derived from corn, wheat, rice, potato, tapioca, or high amylose starch. Sources of starch which may be used also include flours from grains such as corn, wheat, durum wheat, rice, barley, oat, or rye, and mixtures thereof. In preferred embodiments, finely ground or powdered cookies or crackers, or ground cookie-like or cracker-like products are employed with substantially no additional, plasticizable, gelatinized matrix materials.

Additional matrix components which may be used include solid components which are substantially non-plasticizable at temperatures lower than the decomposition temperature of the heat sensitive encapsulant. Exemplary of such optional, substantially non-plasticizable matrix components are at least substantially non-gelatinized starch, carbohydrates which have a lower molecular weight than starches, fiber, or other inert materials, such as cellulose, or hemi-cellulose. The lower molecular weight matrix components tend to dissolve more readily than does starch and increase the penetrability or porosity of the matrix. As a result, access by the dissolution medium, such as water or acid, to the encapsulant is increased thereby permitting quicker release of the encapsulant from the matrix material. Examples of carbohydrates other than starch which may be used are sugars, such as mono- and di-saccharides, and starch hydrolyzate products such as dextrins or syrups with dextrose equivalent values (DE values) ranging from about 2 to about 99, or from about 5 to 98, and mixtures thereof

Additional ingredients which may be used to control the release properties of the final product may be a hydrophobic agent for slowing down the rate of release of the encapsulant. Exemplary of components which may be added to affect the hydrophobicity of the matrix composition include fats, oils, waxes, fatty acids, emulsifiers, such as mono- or di-glycerides, modified starches from plant sources that possess hydrophobic properties that are obtained via either physical or chemical modification, and mixtures of hydrophobic components. Plant lipids or synthetic lipids with melting points up to about 65° C. may, for example, be employed as a hydrophobic agent. The hydrophobic components increase the hydrophobicity of the matrix and help to prevent or delay penetration of water or gastric juice into the matrix by repelling water or aqueous acids, thereby delaying the release of the encapsulant into the surrounding media.

Additional components which may be used to delay or prevent a fast release of the encapsulant from the matrix are components or agents which have a high water binding capacity. The agents may have a water binding capacity or water holding capacity which is greater than the water binding capacity of the free-flowing mixture, such as ground cookies. The high water binding capacity component may bind water which penetrates the particles, or prevent the water from dissolving the matrix, thereby preventing or delaying the release of the encapsulant from the matrix. Exemplary of high water binding capacity agents which may be used in the present invention are protein from animal sources such as gelatin, casein, and protein from sources such as wheat, soy, corn, or other grains, and hydrocolloids such as carrageenans, alginates, xanthan gum, gum arabic, guar flour or guar gum, agar, tragacanth, karaya, locust bean gum, pectin, soluble fiber, insoluble fiber and the like. Exemplary proteins from grains which may be used are gluten, vital wheat gluten, zein, and soy protein concentrate. The proteins from plant sources may also be used to increase the tolerable addition of lipids within the matrix composition and thereby indirectly increase the hydrophobicity of the matrix. The high water binding capacity components may be used alone or mixtures thereof may be employed.

Process compatible additional components to facilitate processing, or to improve sensory attributes such as the taste, texture, aroma, color, appearance, or hydration behavior of the final pellets which may be employed include: flavors, sodium chloride, nonfat dry milk, whey protein, high fructose corn syrup, leavening agents, lipids, such as oils or fats, chocolate liquor, chocolate, cocoa powder, compound coatings, concentrated fruit Juice, or particulates, such as ground nuts or almonds. The water may be pH adjusted to obtain a good tasting product. The addition of vegetable oil during mixing has been found useful to obtain a smooth continuous dough phase and it facilitates forming of the dough and cutting into discrete particles without sticking.

The additional components or ingredients, such as those used to control the rate of release of the encapsulant may be used in amounts up to about 70% by weight, preferably from about 5% by weight to about 50% by weight, most preferably from about 10% by weight to about 35% by weight, based upon the weight of the free-flowing mixture, such as ground-up cookies.

The encapsulant may be in solid or liquid form for inclusion in the matrix compositions and encapsulated products of the present invention. Active components which may be encapsulated or embedded in the matrixes in accordance with the present invention include pharmaceutical compositions or compounds, nutraceutical compositions or compounds, nutritional components, or biologically active components, flavorants, and fragrances.

The pharmaceutical compounds or compositions and biologically active compositions may, for example, include antibiotics, analgesics, vaccines, antiinflammatory agents, antidepressants, anti-viral agents, anti-tumor agents, enzyme inhibitors, formulations containing zidovudine, macromolecular polypeptides, aromatic nitro and nitroso compounds and their metabolites useful as anti-viral and anti-tumor agents, HIV protease inhibitors, viruses, and steroids, compositions to promote growth such as hormones, or other growth stimulating agents, mixtures thereof, and the like.

Nutraceutical components may include components which promote health or prevent disease or enhance well-being such as antioxidants, phytochemicals, hormones, vitamins such as Vitamins A, B1, B2, B6, B12, C, D, E, K, pantothenate, folic acid, pro-vitamins, minerals such as calcium, selenium, magnesium salts, available iron, and iron salts, microorganisms such as bacteria, such as live lactobacilli, fungi, and yeast, prebiotics, probiotics, trace elements, essential and/or highly unsaturated fatty acids such as omega-3 fatty acids, and mid-chain triglycerides, nutritional supplements, enzymes such as amylases, proteases, lipases, pectinases, cellulases, hemicellulases, pentosanases, and phytases, pigments, oligopeptides, dipeptides, and amino acids, and mixtures thereof.

Exemplary of the active components which may be encapsulated or embedded in accordance with the present invention are: acepromazine, acetaminophen, acetohexamide, acetohydroxamic acid, acetylcholine, acetylcysteine acyclovir, albendazole, alclometasone dipropionate, allopurinol, alprazolam, alprostadil, amcinoide, amantadine, amdinocillin, amikacin amiloride, aminocaproic acid, aminophylline, aminosalicylate, aminosalicylic acid, amitriptyline hydrochloride, ammonium chloride, amobarbital, amodiaquine hydrochloride, amoxapine, amoxicillin, amphetamine sulfate, amphotericin, ampicillin amprolium, acetazolamide acetyldigoxin, acetylsalicylic acid, anileridine, anthralin, antipyrine, antivenin, apomorphine, apraclonidine, ascorbic acid, aspirin, acromycin atropine, amoxycillin anipamil, azaperone azatadine maleate, azathioprine, azithromycin, aztreonam, bacampicillin, bacitracin, baclofen, barium salts, beclomethasone diproionate, belladonna extract, bendroflumethiazide, benoxinate hydrochloride, benzethonium chloride, benzocaine, benzonatate benzthiazide, benztropine mesylate, betaine, betamethasone, betaxolol, betanechol chloride, biotin, biperiden, bisacodyl, bismuth, botulism antitoxin, bromocriptine mesylate, bromodiphenhydramine hydrochloride, bumetanide, bupivacaine, busulfan butabarbital sodium, butalbital, combinations of butalbital, caffeine and aspirin and codeine, beta-carotene, calcifediol, calcium carbonate, calcium citrate, calcium salts, candicidin, captopril, carbachol, carbamazepine, carbenicillin indanyl sodium, carbidopa, carbinoxamine maleate, carboprost tromethamine, carboxymethyl cellulose, carisoprodol, casanthranol, cascara, castor oil, cefaclor, cefadroxil, cefamandole nafate, cefazolin, cefixime, cefoperazone, cefotaxime, cefprozil, ceftazidime, cefuroxime axetil, cephalexin, cephradine, chlorambucil, chloramphenicol, chlordiazepoxide, chloroquine phosphate, chlormadinone acetate, chlorothiazide, chlorpheniramine maleate, chloroxylenol, chlorpromazin, chlorpropamide, chlorprothixene, chlorprothixene, chlortetracycline bisulfate, chlortetracycline hydrochloride, chlorthalidone, chlorzoxazone, cholecalciferol, cholera vaccine, chromic chloride, chymotrypsin, cimetidine, cinoxazin, cinoxate, ciprofloxacin, cisplatin, clarithromycin, clavulanate potassium, clemastine fumarate, clidinium bromide, clindamycin hydrochloride, -palmitate and -phosphate, clioquinol, clofazimine, clofibrate, clomriphene citrate, clonazepam, cinnarizine, clonidine hydrochloride, clorsulon, clotrimazole, cloxacillin sodium, cyanocobalamin, cocaine, coccidioidin, cod liver oil, codeine, colchicine, colestipol, corticotropin, corisone acetate, cyclacillin, cyclizine hydrochloride, cyclobenzaprine hydrochloride, cyclophosphamide, cycloserine, cyclosporine, cyproheptadine hydrochloride, cysteine hydrochloride, danazol, dapsone, dehydrocholic acid, demeclocycline, desipramine, desoximetasone, desoxycorticosterone acetate, dexamethasone, dexchlorpheniramine maleate, dexpanthenol, dextroamphetamine, dextromethorphan, diazepam, diazoxide, dibucaine, dichlorphenamide, dicloxacillin sodium, dicyclomine, dienestrol, diethylpropion hydrochlorid, diethylstilbestrol, diflunisal, digitalis, dicoumarol, digitoxin, digoxin, dihydroergotamine, dihydrostreptomycin, dihydrotachysterol, dihydroxyaluminium amino acetate, dihydroxyaluminium sodium carbonate, diltiazem hydrochloride, dimenhydrinate, dimercaprol, diphenhydramine hydrochloride, diphenoxylate hydrochloride, diphteria antitoxin, dipyridamole, disopyramide phosphate, disulfiram, dobutamine hydrochloride, docusate calcium, docusate sodium, dopamine hydrochloride, doxepin hydrochloride, doxycycline, doxycycline hyclate, doxylamine cuccinate, dronabinol, droperidol, drotaverine, dydrogesterone, dyphylline, guaifenesin, enalapril maleate, analaprilat, ephedrine, epinephrine, equilin, ergocalciferol, ergoloid mesylates, ergonovine maleate, ergotamine tartrate, erythrityl tetranitrate, erythromycin, estradiol, estriol, estrogene, estrone, estropipate, ethcrynic acid, ethambutol hydrochloride, ethchlorvynol, ethinyl estradiol, ethionamide, ethopropazine hydrochloride, ethotoin, ethynodiol diacetate, etidronate disodium, etoposide, eugenol, famotidine, fenoprofen, ferrous fumatate, ferrous gluconate, ferrous sulfate, flucytosine, fludrocortisone acetate, flunisolide, fluocinolone acetonide, fluocinonide, fluorescein sodium, fluorometolone, fluorouracil, fluoxymesterone, fluphenazine, flurandrenolide, flurazpam, flurbiprofen, folic acid, furazolidone, flunitrazepam, furosemide, gemfibrozil, gentamicin, gentian violet, glutarate, glutethimide, glycopyrrolate, chorionic gonadotropin, gramicidin, griseofulvin, guaifenesin, guanabenz, guanadrelsulfate, halazone, haloperidol, haloprogin, halothane, heparin calcium, hepatitis virus vaccine, hetacillin potassium, hexylresorcinol, histamine phosphate, histidine, homatropine, histoplasmin, hydralazine hydrochloride, hydrochlorothiazide, hydrocodone bitartrate, hydrocortisone, hexobarbital, hydroflumethiazide, hydromorphone hydrochloride, hydroquinone, hydroxocobalamin, hydroxyamphetamine, hydroxychloroquine sulfate, hydroxyprogesterone caproate, hydroxyurea, hydroxine hydrochloride, hydroxine pamoate, hyoscyamine, hyoscyamine sulfate, ibuprofen, ifosfamide, imipramide, imipramide hydrochloride, indapamide, indomethacin, insulin, inulin, iocetamid, iodoquinol, iohexol, iopamidol, ipecac, ipodate calcium, ipodate sodium, isocarboxacid, isoetharine hydrochloride, isoflurane, isoniacid, isopropamide iodine, isoproterenol hydrochloride, isosorbide dinitrate, isotretenoin, isoxsuprine hydrochloride, kanamycin sulfate, ketoprofen, ketoconazole, labetalol hydrochloride, lanolin, leucine, leucovorin calcium, levamisole hydrochloride, levocarnithine, levodopa, levonorgestrel, levorphanol tartrate, levothyroxine sodium, lidocaine, lincomycin hydrochloride, lindane, liothyronine sodium, liotrix, lisinopril, lithium carbonate, loperamide hydrochloride,

loracarbef, lonetil, lorazepam, lovastatin, loxapine, lysine, mafenide acetate, magaldrte, magnesium carbonate, magnesiumchloride, magnesium gluconate, magnesium oxide, other magnesium salts, malathinon, manganese salts, manganese, maprotiline hydrochloride, mazindol, measle virus vaccine, mebendazole, mebrofenin, mecamylamine hydrochloride, meclizine hydrochloride, meclocycline, meclofenamate sodium, medroxyprogesterone acetate, mefenamic acid, megestrol acetate, meglumine, melphalan, menadiol sodium diphosphate, menadione, menotropine, meperidine, mephenytoin, mephobarbital, meprednisone, meprobamate, mercaptopurine, mesoridazine besylate, mestranol, metaproterenol sulfate, metaraminol bitartrate, methacycline hydrochloride, methadone hydrochloride, methamphetamine hydrochloride, methazolamide, methdilazine, methenamine, methicillin sodium, methimazole, methionine, methocarbamol, methotrexate, methoxsalen, methoxyflurane, methsuximide, methyclothiazide, methylbenzethonium chloride, methyldopa, methylergonovine maleate, methylphenidate hydrochloride, methylprednisolone, methyltestosterone, methysergide maleate, metoclopramide, metolazone, meoprolol tartrate, metronidazole, metyrapone, metyrosine, mexiletine hydrochloride, mexiletine hydrochloride, miconazole, minocycline hydrochloride, minoxidil, mitomycin, mitotane, molindone hydrochloride, monobenzone, morphine sulfate, mupirocin, medazepam, mefruside, methandrostenolone, methylsulfadiazine, nadolol, nafcillin, nafcillin sodium, nalidixic acid, nalorphine, naloxone, nandrolone decanoate, nandrolone phenpropionate, naproxen, natamycin, neomycin, neomycin sulfate, neostimine bromide, niacin, nitrofurantoin, nalidixic acid, nifedipine, nitrazepam, nitrofurantoin, nitroglycerine, nitromerson, nizatidine, nonoxynol 9, norethindrone, norethindrone acetate, norfloxacin, norgestrel, nortriptyline hydrochloride, noscapine, novobiocin sodium, nystatin, opium, oxacillin sodium, oxamniquine, oxandrolone, oxazepam, oxprenolol hydrochloride, oxtriphylline, oxybenzone, oxybutynin chloride, oxycodone hydrochloride, oxycodone, oxymetazoline hydrochloride, oxymetholone, oxymorphone hydrochloride, oxyphenbutazone, oxytetracycline, padimate, panreatin, pancrelipase, papain, panthenol, papaverin hydrochloride, parachlorophenol, paramethasone acetate, paregoric, paromomycin sulfate, penicillamine, penicillin, penicillin derivatives, pentaerythritol tetranitrate, pentazocine, pentazocine hydrochloride, pentazocine salts, pentobarbital sodium, perphenazine, pertussis, phenacemide, phenazopyridine hydrochloride, phendimetrazine tartrate, phenelzine sulfate, phenmetrazine hydrochloride, phenobarbital, phenophtalein, phenoxybenzamine hydrochloride, phentermine hydrochloride, phenylalanine, phenylbutazone, phenylephrine hydrochloride, phenylpropanolamine hydrochloride, physostigmine, phytonadione, pilocarpine, pimozide, pindolol, piperazine, piroxicam plicamycin, poliovirus vaccine inactivated, polycarbophil, polymycin b sulfate, polythiazide, potassium chloride, potassium citrate, potassium cluconate, potassium iodine, potassium sodium tartrate, povidone iodine, pralidoxime chloride, pramoxine hydrochloride, pramezam, prazepam, praziquantel, prazosin hydrochloride, prazosin hydrochloride, prednisolone, prilocaine, primaquine, primidone, probenecid, probucol, procainamide hydrochiorid, procaine hydrochloride, procarbacine hydrochloride, prochlorperazine, prochlorperazine maleate, procyclidine hydrochloride, progesterone, proline, promazine, promazine hydrochloride, promazine, promethazine, promethazine hydrochloride, propafenone hydrochloride, propantheline, proparacaine hydrochloride, propoxycaine hydrochloride, propoxyphene hydrochloride, propoxyphene napsylate, propanolol hydrochloride, propyliodone, propylthiouracil, propylthiouracil, protriptyline hydrochloride, pseudoephedrine hydrochloride, pumice, pyrantel pamoate, pyrazinamide, pyrethrum extract, pyridostigmine bromide, pyridoxine hydrochloride, pyrilamine maleate, pyrimethamnine, pyroxylin, pyrvinium pamoate, phenacetin, phenytoin, prednisone, uinidine gluconate, quinidine sulfate, rabies vaccine, racepinephrine ranitidine, rauwolfia serpentina, resorcinol, ribavirin, riboflavin, rifampin, ritodrine, rubella virus vaccine, saccharin, saccharin sodium, salicylamide, salicylic acid, salsalata, scopolamine, secobarbital sodium, selenius acid, selenium sulfate, sennasenrne, simethicone, sodium ascorbate, sodium bicarbonate, sodium fluoride, sodium gluconate, sodium iodide, sodium lactate, sodium nitrite, sodium ditroprusside, sodium salicylate, spironolactone, stannozolol, streptomycin, sucralfate, sulfacetamide, sulfadiazine, reserpine, sulfadioxine, sulfamerazine, sulfamethazine, sulfamethizole, sulfamethoxazole, sulfamethoxydiazine, sulfapyridin, sulfasalazine, sulfaperin, sulfathiazole, sulfisoxazole, sulfinpyrazone, sulindac, suprofen, stilains, tamoxifen citrate, temacepam, terbutaline sulfate, terfenadine, terpin, testolacton, testosterone, tolazamide, tolbutamide, tetracaine, tetracycline, tetrahydrocycline, theophylline, thiabendazole, thiamine hydrochloride, thiamin, thiamylal, thiethylperazine thimerosal, thioguanine, thioridazine hydrochloride, thistrepton, thiotepa, thiothixene, threonine, thyroid, ticarcillin, timolol, tioconazole, titaniumdioxide, tolazamide, tolbutamide, tolmetin, tolnaftate, trazodone hydrochloride, tretinoin, triacetin, triamcinolone, triamterene, triazolam, trichorfon, trichlormethiazide, trientine hydrochloride, trifluoperazine hydrochloride, triflupromazine, trihexyphenidyl hydrochloride, trimeprazine tartrate, trimethadione, trimethobenzamide hydrochloride, trimethoprim, trioxsalen, tripelennamine, triprolidine, trisulfapyrimidine, tropicamide, trypsin, tryptohan, tuberculin, tyloxapol, tyropanoate sodium, tyrosine, tyrothricin, thyrothricin bethamethasone, thiotic acid, sotalol, salbutamol, norfenefiine, silymarin, dihydroergotamine, buflomedil, etofibrate, indometacin, urea, valine, valproic acid, vancomycin hydrochloride, vasopressin, verapramil, vidarabine, vinblastine, vincristine, vitamins, warfarin, yellow fever vaccine, zinc acetate, zinc carbonate, zinc chloride, zinc gluconate, beta acetyl digoxin, piroxicam, haloperidol, ISMN, amitriptylin, diclofenac, nifedipine, verapamil, pyritinol, nitrendipin, doxycycline, bromhexine, methylprdnisolone, clonidine, fenofibrate, allopurinol, pirenyepine, levothyroxin, tamoxifen, metildigoxin, o-(beta-hydroxyethyl)rutoside, propicillin, aciclovir mononitrate, paracetamol, naftidrofuryl, pentoxifilline, propafenone, acebutolol, L-thyroxin, tramadol, bromocriptine, loperamide, ketotifen, fenoterol, cadobelisate, propanolol, enalaprilhydrogen maleate, bezafebrate, ISDN, gallopamil, xantinol nicotinate, digitoxin, flunitrazepam, bencyclane, dexapanthenol, pindolol, lorazepam, diltiazem, piracetam, phenoxymethylpenicillin, furosemide, bromazepam, flunarizin, erythromycin, metoclopramide, acemetacin, ranitidin, biperiden, metamizole, doxepin, dipotassium chloroazepate, tetrazepam, estramustine phosphate, terbutaline, captopril, maprotiline, prazosin, atenolol, glibenclamide, cefaclor, etilfrine, cimetidine, theophylline, hydromorphone, ibuprofen, pnimidone, clobazam, oxaceprol, medroxyprogesterone, flecainid, pyridoxal 5

phosphate glutaminate, hymechromone, etofylline clofibrate, vincamine, cinnarizine, diazepam, ketoprofen, flupentixol, molsimine, glibornuride, dimetinden, melperone, soquinolol, dihydrocodeine, clomethiazole, clemastine, glisoxepide, kallidinogenase, oxyfedrine, baclofen, carboxymethylcysteine, thioridazine, betahistine, L-tryptophan, murtol, bromelaine, prenylamine, salazosulfapyridine, astemizol, sulpiride, benzerazide, dibenzepine, acetylsalicylic acid, miconazol, nystatin, ketoconazole, sodium picosulfate, coltyramine, gemfibrocil, rifampicin, fluocortolone, mexiletin, amoxicillin, terfenadrin, mucopolysaccharide polysulfade, triazolam, mianserin, tiaprofenic acid, amezinium metilsulfate, mefloquine, probucol, quinidine, carbamazepine, L-aspartate, penbutolol, piretanide, aescin amitriptyline, cyproterone, sodium valproinate, mebeverine, bisacodyl, 5-aminosalicylic acid, dihydralazine, magaldrate, phenprocoumon, amantadine, naproxen, carteolol, famotidine, methyldopa, auranofine, estriol, nadolol, levomepromazine, doxorubicin, medofenoxate, azathioprine, flutamide, norfloxacin, fendiline, prajmalium bitartrate, lipid derivatives of phosphonatides, amphiphilic polymers, adenosine derivatives, sulfated tannins, monoclonal antibodies, and metal complexes of water soluble texathyrin.

The amount of the active component or encapsulant which is incorporated into the products of the present invention may be such so as to provide or deliver an effective amount, such as a pharmaceutically effective amount or a nutraceutically effective amount of the active component at its intended location, such as the small intestine. Exemplary amounts of the active component or encapsulant which may be encapsulated or embedded into the matrix may be from about 1% by weight to about 85% by weight, preferably from about 3% by weight to about 50% by weight, most preferably from about 5% by weight to about 30% by weight, based upon the weight of the free-flowing mixture, such as ground cookies. In embodiments of the invention, the amount of the active component or encapsulant may be up to about 80% by weight, preferably up to about 50% by weight, most preferably up to about 20% by weight of the plasticizer, such as oil.

In embodiments of the invention, the encapsulants and/or the edible, chewable matrix composition may be coated to provide additional protection against oxygen, to provide mechanical stability against abrasion, or to control release of the encapsulant without negatively influencing the chewable texture of the matrix composition. Film-building or film-forming substances which may be used to coat encapsulants prior to admixing with the free-flowing mixture and prior to incorporation into the matrix include commonly used coating materials. Exemplary of coating materials which may be employed are zein, pectin, shellac, gelatin, gluten, fats, oils, waxes, emulsifiers, native or modified starch, chitosan, chitin, and mixtures thereof These film-building or film-forming substances may also be used to coat the extruded, particulate chewable product. Pretreatment of the encapsulant by coating it with a film forming substance such as a high melting fat or wax, or with an emulsifier such as glycerin monostearate, or the like, tends to prevent unwanted interaction between an encapsulant and the matrix. The encapsulants and the extrudate particles may be coated with film-forming amounts of the substances in aqueous or alcoholic solutions, or oleaginous compositions. The encapsulants may be pre-encapsulated or pre-coated using any conventional encapsulation or coating method which does not thermally destroy the encapsulant.

In embodiments of the invention pellets may be coated in a two step coating process. After discrete particles have been cut at an extrusion die, the substantially undried pellets may be coated with a first component of a composite coat, such as an acetic-acid-chitosan-solution. After this step a second coat may be applied using a gel forming counter ion, such as a polyphosphate solution, that causes the chitosan to gel and form a chitin coat. The second ion may also be provided by a pectin and the resulting composite coat may then be a pectin-chitin coacervate.

The film-forming substances or coatings may also contain additional components that protect the particulates or pellets, or encapsulant, from the influence of light, such as titanium dioxide, or cocoa-based products. The coatings may also contain anti-oxidants to protect the pellets or encapsulants from the influence of oxygen or air.

In accordance with embodiments of the present invention, the thickness of the coating upon the encapsulant may be used to control the rate of release of encapsulant once the dissolving media, such as water, reaches the encapsulant. For example, increasing the thickness of the coating on the encapsulant slows its rate of release into the media. Also, increasing the thickness of the coating on the extrudate or pellet delays release of the encapsulant from the matrix material. In embodiments of the invention, the amount of coating may range from about 0.5% to about 50% by weight, based on weight of the total product, depending upon the desired release of the encapsulant.

In accordance with the method of the present invention, all of the ingredients may be admixed together at a temperature of about 5° C. to about 50° C., for example about 30° C. to obtain a formable mixture or dough. Mixing or dough temperatures substantially higher than about 50° C. are undesirable, because any fat or oil in the formula tends to separate, or the heat sensitive substances to be encapsulated and embedded would be destroyed. Temperatures much lower than room temperatures, such as for example 0° C. are for most purposes impractical but may be employed in special applications. In embodiments of the invention, the temperature may be adjusted by external heating, below 50° C. so as to facilitate forming and enable cutting without the material sticking to the cutter.

In embodiments of the present invention, external heating of the ingredients during their admixture is not required. For example, where ground cookies is used as the free-flowing matrix material, heating of the ground cookies and water to cook or gelatinize the starch is not needed to obtain a formable dough which when dried to a shelf-stable moisture content provides a chewable texture.

The admixing of the ingredients is conducted under low shear mixing conditions without substantially destroying or decomposing the matrix material or encapsulant. An overall quantitative measure of the shear used inside an extruder, for example, is the specific mechanical energy input. In embodiments of the present invention, the specific mechanical input during admixing of the ingredients to obtain a formable mixture or dough may be below about 150 Wh/kg, preferably below about 100 Wh/kg, and most preferably below about 50 Wh/kg.

In embodiments of the invention, the pressure under which the formable mixture or dough may be formed may range from about 1 bar to about 100 bars. It is preferably formed into individual shapes at pressures of about 5 bars to about 60 bars.

The optional one or more additional ingredient or component, such as the hydrophobic component, or high

water binding capacity component for controlling the release properties of the final product, may be dry blended or preblended with the free-flowing matrix material such as ground cookies. In other embodiments of the invention, the additional component for controlling the release properties may be added separately.

The encapsulant may be added either during the dough mixing process or it may be added into the dough after it is produced. After addition and admixing of the encapsulant, the dough may be compressed and shaped into discrete shapes.

In embodiments of the invention, a dough comprising all ingredients may be made using conventional batch or continuous mixers. Subsequently, the dough, which may be a crumbly dough may be fed into a single screw extruder. The single screw extruder presses the dough against a die plate and plasticizes the crumbs into a continuous dough phase which may then be pressed through an extrusion die and subsequently cut into individual particulates.

In other embodiments of the invention, the dough can be made continuously and using a continuous mixer or extruder alone. Twin screw extruders or co-rotating twin screw mixers may be advantageously used which enable the steps of continuously mixing the dough and subsequently extruding the dough through an extrusion die plate. Co-rotating intermeshing twin screw extruders, such as those available from Buhler, Switzerland, Clextral France, Werner and Pfleiderer Germany, APV England or Wenger USA, or a Co-Kneader, available from Buss, Switzerland may be employed.

For feeding solid components to an extruder, conventional solids feeding devices such as a volumetric or gravimetric feeder may be used. Liquid injection nozzles may be used for injecting liquid active components or solutions, dispersions, emulsions or suspensions. In embodiments of the invention, a side feeder and liquid injection nozzles may be employed. If an injection nozzle is used, the pressure for injecting the liquid encapsulant should be sufficiently higher than the pressure in the extruder so that the encapsulant can be injected into the extruder barrel. For example, if the pressure of the plasticized mass inside the extruder is 10 bars, the injection pressure may be about 2 to about 5 bars higher, i.e. 12 to 15 bars.

In embodiments where an encapsulant is pre-coated with a film-building material or coating material, the coating material may be applied in conventional manner such as by spraying or enrobing using conventional coating equipment. Commercially available pre-coated active ingredients, such as precoated minerals or vitamins may be employed.

The admixing of the added active ingredients or encapsulants inside the extruder may be accomplished by using an appropriate extrusion screw configuration for achieving low shear mixing. For example, a combination of alternating small pitch conveying elements with distributive mixing elements, that are staggered at an angle to each other for providing axially oriented leakage flow inside the extruder barrel may be employed. The combination of alternating conveying elements with distributive mixing elements cause the material flow to be continuously interrupted without shearing of the mass thus resulting in mixing of the material at low mechanical energy input.

In other embodiments of the invention, other extruder screw configurations may be used that facilitate low shear distributive mixing, such as screw elements of the type ZME, TME, SME, and so-called IGEL elements commercially available from Werner and Pfleiderer.

The total length of the distributive mixing section may be about 3 to 12 l/d, preferably about 4 to 6 l/d to sufficiently admix and distribute and embed or encapsulate the added active components in the matrix.

The at least substantially homogeneous mixture of matrix material and added active ingredient or encapsulant may then be conveyed towards an extruder die plate. The conveying may be achieved by the use of low pitch extruder screw conveying elements which build up sufficient pressure prior to extruding the mix so that it can be forced through the apertures in the die plate. Another function of the low pitch elements is that they increase the degree of fill inside the last extruder barrel section. The increased degree of fill enables control of the temperature profile of the mix inside the extruder barrel for achieving optimum viscosity adjustment and extrusion through the subsequent die openings.

The dough or crumbly mass or mix may be extruded or pressed through extrusion dies having aperture diameters of from about 0.3 mm to about 5 mm, preferably from about 0.5 mm to about 3 mm, for example from about 0.5 mm to about 1 mm. The diameter of the extrudate rope and product may be larger than the diameter of the die apertures due to deformation or swelling as the composition exits the die. The increase in diameter upon exiting the die may occur without substantial development of an expanded, puffed, foamy, or cellular structure. The extruded rope may have a cross-sectional diameter of from about 0.5 mm to about 7 mm, preferably from about 0.5 mm to about 5 mm, most preferably from about 0.5 mm to about 3 mm.

The extrudate rope may be cut at the die face using a rotating cutter, pelletizer, or rotating knives. In other embodiments, the extrudate rope may be cut away from the die using conventional cutting or forming means for producing pellets or tablets. The cut pieces, pellets, or tablets, may have a length:diameter ratio (l/d ratio) of about 0.5 to 10, preferably about 1.

In accordance with the process of the present invention, the particle size may be varied to control the surface to volume ratio of the pellets or pieces for achieving a desired controlled release of the encapsulant. The particle size may be varied, for example, by the use of different diameters for the extrusion die openings. Particle size may also be varied by the use of a variable speed cutter either at the die plate at the end of the extruder or away from the extruder after the ropes have been conveyed for a short distance. By varying the speed of the cutter, the size of the cut pieces may be varied for a given extruder throughput. The use of a variable cutter which is spaced a short distance from the die plate, for example, between about 0.5 meters to about 5 meters permits further surface cooling, further surface drying, and reduced stickiness to provide better cutting of the ropes into pellets.

In producing products for human or animal consumption, variation of particle size to control the surface to volume ratio of the pellets is critical for achieving a controlled release of the encapsulant during passage of the pellets or particles through the mouth, the stomach, and the intestine. Variation of particle size is also critical for controlling the residence time of the pellets inside the stomach. For example, particles smaller than 1 mm pass through the stomach or intestine faster than would particles larger than for example 2.5 mm.

After cutting, the resulting pieces or pellets may be dried to a sufficiently low moisture content which assures a sufficiently prolonged storage stability or shelf life. For example, the pellets may be dried to achieve a storage

stability or shelf life of at least about six months, preferably at least about twelve months, most preferably at least about thirty-six months. In embodiments of the present invention, the drying may be performed using conventional drying equipment using drying temperatures which do not adversely affect the thermal stability of the encapsulants. Exemplary drying temperatures may range from about 10° C. to about 50° C., for example about 30° C. The drying may be conducted to achieve a moisture content of less than about 30% by weight, preferably less than about 12% by weight, most preferably less than about 10% by weight, for example less than about 8% by weight.

The product may be dried using a conventional fluidized bed or other conventional drying means. The product may be optionally coated after drying using conventional coating equipment such as coating pans, coating drums, or spray devices.

In embodiments where film-building substances or coatings are applied to the particles or pellets, conventional spray nozzles may be located close to the die for spraying an aqueous or alcoholic solution of the film-building substances onto the cut pieces as they fall downwardly from the extruder die. In other embodiments, the film-building substances may be applied after drying of the pellets. For example, the film-building substances may be applied using spray nozzles, conventionally known fluid bed coating apparatus, or other conventional coating apparatus and methods. If the application of the film-building substances increases the moisture content above a shelf stable level, the water or other volatile media may be removed from the surface of the particles by additional drying.

In embodiments of the present invention, the extruded pieces or pellets may be compressed in conventional tablet presses to obtain compressed versions of the extruded pellets.

In other embodiments of the present invention, the mixture may be extruded or formed into bars or into a rope which may be cut into food bar-sized pieces. The mixture may also be extruded through a sheeting die into a sheet. The extruded sheet may then be cut or molded into individual pieces, such as bars, snack-sized pieces, tablets, or disks, using a rotary die or rotary cutter, or reciprocating cutter or counterrotating drums conventionally known as agglomeration drums or tableting drums.

The products of the present invention may possess a chewable texture, like that of streusel or chewable vitamin pills with a cookie-like taste. They may comprise food bar or snack-sized pieces, or they may comprise discrete particles which may be spherical, lens-shaped, or flat discs having diameters of from about 0.5 mm to about 7 mm, preferably from about 0.5 mm to about 5 mm, most preferably from about 1 mm to about 3 mm, exclusive of any optional exterior film-building substances or coatings. In embodiments of the invention, the particles of the invention may be in the form of tablets with diameters of up to about 10 mm. The length-to-diameter ratio (l/d) of the particles may be from about 0.1 to about 10, for example about 0.5 to about 2, preferably about 1. The particles are generally uniform in size, non-glassy, and granular to increase palatability to humans and animals in a substantially compact form that is easy to swallow with or without chewing. The products of the invention are non-expanded, generally not leavenable, and exhibit a non-puffed, substantially non-cellular, non-glassy structure. The starch component of the matrices may be substantially ungelatinized, and not substantially destructurized or dextrinized. Exemplary specific densities of the products of the present invention are between about 800 g/liter and about 1500 g/liter (about 0.8 to about 1.5 g/cm^3).

The encapsulated products of the present invention may be incorporated with or without grinding into foods intended for human or animal consumption such as baked goods, for example, bread, wafers, cookies, crackers, pretzels, pizza, and rolls, ready-to-eat breakfast cereals, hot cereals, pasta products, snacks such as fruit snacks, salty snacks, grain snacks, and microwave popcorn, dairy products such as yoghurt, cheese, and ice cream, sweet goods such as hard candy, soft candy, and chocolate, beverages, animal feed, pet foods such as dog food and cat food, aqua-culture foods such as fish food and shrimp feed, and special purpose foods such as baby food, infant formulas, hospital food, medical food, sports food, performance food or nutritional bars, or fortified foods, food preblends or mixes for home or food service use, such as preblends for soups or gravy, dessert mixes, dinner mixes, baking mixes such as bread mixes, and cake mixes, and baking flour.

In preferred embodiments, the active encapsulant is either a live microorganism, enzyme, micronutrient, trace element, nutraceutical component, biologically active material or a combination thereof The encapsulated product may be redispersed as a liquid, or as a solid for human food, animal feed, or pharmaceutical purposes. The products of the present invention may be used as or incorporated into foods for special purposes, such as performance foods, mood foods, medical foods, nutritional snacks or supplements, sport foods such as power bars, baby foods, toddler foods, infant foods, or foods for pharmaceutical purposes or other dietetic purposes. The discrete particulates or granules of the present invention may be used as a topping for breakfast cereals, snacks, soups, salad, cakes, cookies, crackers, puddings, desserts or ice cream. They may also be used as a granular ingredient for yogurts, desserts, puddings, custards, ice cream or other pasty or creamy foods. Regularly sized pieces may be individually packaged or used as nutritional snacks or, for example added to or formed into nutritional food in bar form.

The present invention is further illustrated by the following non-limiting examples where all parts, percentages, proportions, and ratios are by weight, and all temperatures are in ° C. unless otherwise indicated:

EXAMPLE 1

Production of Good Tasting and Chewable Pellets Having Encapsulated Live Microorganism

In this example 10 kg cookies (Leibnitz Keks, Bahlsen, Germany) were ground with a hammer mill into a flour having a particle size of 100% less than 1 mm. This flour was fed at a rate of 4 kg/h into a twin screw extruder (Werner & Pfleiderer, ZSK22). A mix of water and citrus juice (ratio 7:1) was fed at a rate of 0.8 kg/h into the same extruder. 0.188 kg of *Lactobacillus Acidophilus* was preblended with 0.375 kg vegetable fat (BISKIN) and 0.188 kg vegetable oil and the preblend was fed at a rate of 0.750 kg/h into a subsequent barrel of the same extruder. The barrel temperature of the extruder was kept at 20° C. The extruder conditions were:

rpm	150 revolutions per minute
Pressure	45 bar

-continued

Product Temperature	31° C.
Die	20 × 1 mm diameter

The product was cut into individual pellets at the die face with a rotating knife. The pellets were dried at 30° C. in a convection batch dryer for 1 hr to 5.9% moisture. The pellets containing live Lactobacilli exhibit a pleasant taste and chewable texture.

EXAMPLE 2

Coating With Thin Additional Coat

A portion of the pellets made in Example 1 were coated with a 25% shellac/alcohol solution to obtain a 5% shellac coat based on the total weight of the total product. These pellets containing live lactobacilli exhibit a pleasant taste and chewable texture, that was slightly harder than the uncoated sample, but still chewable and eatable.

EXAMPLE 3

Coating With Thick Additional Coat

Another portion of the pellets obtained in Example 1 were coated with a 25% shellac/alcohol solution to obtain a 10% shellac coat based on the total weight of the total product. The obtained pellets containing live lactobacilli exhibit a pleasant taste and chewable texture, that was slightly harder than the 5% coated sample of Example 2, but they were still chewable and eatable.

COMPARATIVE EXAMPLE

Semolina flour was fed at a rate of 4 kg/h into a twin screw extruder (Werner & Pfleiderer, ZSK22). Water was fed at a rate of 1.5 kg/h into the same extruder. 0.18 kg of *Lactobacillus Acidophilus* was preblended with 0.36 kg vegetable fat (BISKIN) and 0.18 kg vegetable oil and the preblend was fed at a rate of 0.72 kg/h into a subsequent barrel of the same extruder. The barrel temperature of the extruder was kept at 20° C.

The extruder conditions were:

rpm	120 revolutions per minute
Pressure	20 bar
Product Temperature	29° C.
Die	20 × 1 mm diameter

The product was cut into individual pellets at the die face with a rotating knife. The pellets were dried at 30° C. in a convection batch dryer for 1 hour to 8.25% moisture. The pellets containing live lactobacilli exhibit a very hard and unacceptable texture to be eaten as such. The texture was comparable to that of uncooked pasta.

What is claimed is:

1. An edible matrix composition that has a chewable texture and that contains at least one component encapsulated by the matrix composition, said matrix composition comprising at least one plasticizer, and a free-flowing particulate mixture which comprises at least one fat, at least one starch, and at least one sugar which have been mixed and heated, said at least one starch having a degree of gelatinization of less than about 50%.

2. An edible matrix composition as claimed in claim 1 wherein said free-flowing mixture is obtained by baking a mixture of said at least one fat, at least one starch, and at least one sugar without substantially gelatinizing said at least one starch to obtain a baked product and then grinding the baked product to obtain said free-flowing mixture.

3. An edible matrix composition as claimed in claim 1 wherein said free-flowing mixture is ground cookies.

4. An edible matrix composition as claimed in claim 1 wherein said plasticizer comprises a fat or oil.

5. An edible matrix composition as claimed in claim 1 wherein said plasticizer comprises water.

6. An edible matrix composition as claimed in claim 1 wherein the free-flowing mixture content is at least about 10% by weight, based upon the weight of said edible matrix composition.

7. An edible matrix composition as claimed in claim 4 wherein the plasticizer content is from about 10% by weight to about 70% by weight, based upon the total weight of said free-flowing mixture and said plasticizer.

8. An edible matrix composition as claimed in claim 1 wherein the plasticizer content is up to about 90% weight, based upon the weight of said edible matrix composition.

9. An edible matrix composition as claimed in claim 1 wherein the plasticizer comprises oil or fat, and the oil or fat plasticizer content is up to 90% by weight, based upon the weight of said edible matrix composition.

10. An edible matrix composition as claimed in claim 9 wherein the melting point of said oil or fat plasticizer is at least about 30° C.

11. An edible matrix composition as claimed in claim 5 wherein the moisture content of the edible matrix composition is less than about 8% by weight, based upon the weight of the matrix composition.

12. An edible matrix composition as claimed in claim 1 wherein said at least one component is selected from the group consisting of biologically active components, pharmaceutical components, nutraceutical components and microorganisms.

13. An edible, chewable matrix composition as claimed in claim 1 wherein the starch content of the matrix composition consists essentially of the starch content of said free-flowing mixture.

14. An edible matrix composition as claimed in claim 3 wherein said ground cookies comprise from about 8% by weight to about 40% by weight shortening or fat, from about 15% by weight to about 40% by weight sugar, and from about 20% by weight to about 75% by weight flour, based upon the weight of the ground cookies.

15. An edible matrix composition as claimed in claim 1 wherein said at least one component comprises live probiotic microorganisms.

16. An edible matrix composition as claimed in claim 15 wherein said at least one component comprises live lactobacilli.

17. An edible matrix composition as claimed in claim 12 which is coated with a coating composition in an amount of from about 0.5% by weight to about 50% by weight, based upon the weight of the coated product.

18. An edible chewable matrix composition as claimed in claim 1 further including at least one flavor or texture enhancing ingredient selected from the group consisting of chocolate, flavors, concentrated juices, fibers, and compound coatings.

19. An edible matrix composition as claimed in claim 18 wherein said at least one flavor or texture enhancing ingredient is selected from the group consisting of emulsifiers, oils and fats.

20. An edible matrix composition according to claim 3 wherein said ground cookies contain at least one component selected from the group consisting of high fructose corn

syrup, maltodextrins, corn syrup, dextrose, lactose, maltose, modified or unmodified starches, leavening agents, non-fat dry milk, full fat dry milk, whey, gluten, soluble and insoluble fiber, nutrients, inulin, hydrocolloids, dry eggs, salt, flavor, emulsifier, cocoa, and cocoa butter.

21. An edible and chewable product comprising:

a) a free-flowing particulate mixture which comprises at least one fat, at least one starch, and at least one sugar which have been mixed and heated, said at least one starch having a degree of gelatinization of less than about 50%, and

b) an encapsulant,

wherein the encapsulant is embedded or encapsulated in a dough made substantially from the free-flowing mixture and a plasticizer, said dough being dried to a shelf-stable moisture content.

22. An edible and chewable product as claimed in claim **21** wherein said free-flowing mixture comprises a flour obtained by grinding cookies.

23. An edible and chewable product as claimed in claim **21** wherein said encapsulant comprises at least one member selected from the group consisting of biologically active components, neutraceutical components, and live microorganisms.

24. An edible and chewable product as claimed in claim **21** wherein said encapsulant is pre-encapsulated.

25. An edible and chewable product as claimed in claim **21** wherein said encapsulant is heat sensitive.

26. An edible and chewable product as claimed in claim **21** wherein said encapsulant has an undesirable taste which is masked by said free-flowing mixture.

27. An edible and chewable product as claimed in claim **21** which is coated with a coating composition.

28. An edible and chewable product as claimed in claim **21** which is a food bar.

29. An edible and chewable product as claimed in claim **21** which is in granular form.

30. A food composition comprising an edible and chewable product as claimed in claim **21** which is selected from the group consisting of ready-to-eat breakfast cereals, snacks, soups, salads, cakes, cookies, crackers, puddings, ice creams, yogurts, puddings, custards, baby foods, medicinal foods, sports bars, and beverages.

31. A food composition as claimed in claim **30** which is a yogurt, pudding, custard, or ice cream wherein said edible and chewable product is in granular form.

32. A food composition as claimed in claim **30** which is a breakfast cereal, snack, soup, salad, cake, cookie, cracker, yogurt, pudding, custard, or ice cream wherein said edible and chewable product is applied as a topping.

33. A food composition as claimed in claim **30** which is a beverage.

34. A food composition comprising a topping which is an edible and chewable product as claimed in claim **30**.

35. A food topping comprising an edible matrix composition as claimed in claim **1** in granular form.

36. An edible, chewable matrix composition comprising:

a liquid plasticizer,

an encapsulant, and

a flour comprising ground cookies,

wherein the starch content of the matrix composition has a degree of gelatinization of less than about 50% so as to provide a chewable, non-glassy texture to the matrix composition, and

wherein the encapsulant is encapsulated by plasticized ground cookies.

37. An edible, chewable matrix composition as claimed in claim **36** wherein said encapsulant comprises at least one member selected from the group consisting of biologically active components, pharmaceutical components, neutraceutical components and microorganisms.

38. An edible, chewable matrix composition as claimed in claim **36** wherein the amount of said ground cookies is at least about 10% by weight of said chewable matrix composition.

39. An edible, chewable matrix composition as claimed in claim **36** wherein the moisture content of the chewable matrix composition is less than about 8% by weight, based upon the weight of the matrix composition.

40. An edible, chewable matrix composition as claimed in claim **36** which is in particulate form.

41. An edible, chewable matrix composition as claimed in claim **36** wherein the starch content of the matrix composition consists essentially of the starch content of said ground cookies.

42. A method for the manufacture of edible products containing an encapsulated component comprising:

a) mixing a free-flowing particulate mixture with a plasticizer to obtain a crumbly mass or dough, said free-flowing mixture comprising a flour made from at least one fat, at least one starch, and at least one sugar which have been mixed and heated so that said at least one starch has a degree of gelatinization of less than about 50%,

b) admixing at least one encapsulant into the crumbly mass or dough,

c) compressing the crumbly mass or dough to obtain a compressed dough,

d) shaping the compressed dough, and

e) separating the shaped dough into individual pieces,

wherein ingredients comprising the free-flowing mixture, plasticizer, and encapsulant are admixed, compressed and shaped at temperatures sufficiently low so as to prevent thermal degradation of the encapsulant and at pressures sufficiently high to enable the formation of coherent pieces.

43. A method as claimed in claim **42** wherein at least said mixing is performed in a continuous mixer or in a twin screw extruder.

44. A method as claimed in claim **42** wherein the compressed dough is shaped by extrusion.

45. A method as claimed in claim **42** wherein said free-flowing mixture is obtained by baking a mixture of said at least one fat, at least one starch, and at least one sugar without substantially gelatinizing said at least one starch to obtain a baked product and then grinding the baked product to obtain said free-flowing mixture.

46. A method as claimed in claim **42** wherein said free-flowing mixture is ground cookies.

47. A method as claimed in claim **42** wherein said plasticizer comprises at least one member selected from the group consisting of fats, oils, and water.

48. A edible matrix composition as claimed in claim **42** wherein said plasticizer comprises fat or oil.

49. A method as claimed in claim **42** wherein said free-flowing mixture comprises a flour obtained by grinding cookies to a particle size of less than about 5 mm.

50. An edible matrix composition as claimed in claim **1**, wherein said at least one component comprises a pharmaceutical composition, a nutraceutical composition, a flavorant, or a fragrance.

51. An edible matrix composition having a chewable texture comprising:

a matrix comprising at least one plasticizer and a free-flowing particulate mixture, said free-flowing mixture comprising at least one fat, at least one starch, and at least one sugar which have been mixed and heated without substantially gelatinizing said at least one starch; and

at least one component encapsulated by the matrix and selected from the group consisting of analgesics, anti-inflammatory agents, anti-depressants, anti-viral agents, anti-tumor agents, enzyme inhibitors, steroids, hormones, HIV protease inhibitors, and mixtures thereof.

52. An edible matrix composition having a chewable texture comprising:

a matrix comprising at least one plasticizer and a free-flowing particulate mixture, said free-flowing mixture comprising at least one fat, at least one starch, and at least one sugar which have been mixed and heated without substantially gelatinizing said at least one starch; and

at least one component encapsulated by the matrix and selected from the group consisting of antioxidants, phytochemicals, vitamins, folic acid, pantothenate, minerals, fatty acids, enzymes, amino acids, and mixtures thereof.

53. An edible matrix composition having a chewable texture comprising:

a matrix comprising at least one plasticizer and a free-flowing particulate mixture, said free-flowing mixture comprising at least one fat, at least one starch, and at least one sugar which have been mixed and heated without substantially gelatinizing said at least one starch; and

an omega-3 fatty acid encapsulated by the matrix.

54. An edible matrix composition according to claim **1**, wherein said at least one starch has a degree of gelatinization of less than about 30%.

55. An edible matrix composition according to claim **1**, wherein said at least one starch has a degree of gelatinization of less than about 15%.

56. An edible matrix composition according to claim **1**, wherein said at least one plasticizer is selected from the group consisting of a sugar solution, juice, alcohol, glycerol, and sorbitol.

57. An edible matrix composition according to claim **1**, wherein said at least one plasticizer is an alcohol.

58. An edible and chewable product according to claim **21**, wherein said at least one starch has a degree of gelatinization of less than about 30%.

59. An edible and chewable product according to claim **21**, wherein said at least one starch has a degree of gelatinization of less than about 15%.

60. An edible, chewable matrix composition according to claim **36**, wherein the starch content of the matrix composition has a degree of gelatinization of less than about 30%.

61. An edible, chewable matrix composition according to claim **36**, wherein the starch content of the matrix composition has a degree of gelatinization of less than about 15%.

62. A method according to claim **42**, wherein said free-flowing mixture comprises a flour made from at least one fat, at least one starch, and at least one sugar which have been mixed and heated so that said at least one starch has a degree of gelatinization of less than about 30%.

63. A method according to claim **42**, wherein said free-flowing mixture comprises a flour made from at least one fat, at least one starch, and at least one sugar which have been mixed and heated so that said at least one starch has a degree of gelatinization of less than about 15%.

64. An edible matrix composition according to claim **51**, wherein said at least one starch has a degree of gelatinization of less than about 50%.

65. An edible and chewable product according to claim **52**, wherein said at least one starch has a degree of gelatinization of less than about 50%.

66. An edible and chewable product according to claim **53**, wherein said at least one starch has a degree of gelatinization of less than about 50%.

67. An edible matrix composition that has a chewable texture, comprising:

a matrix comprising at least one plasticizer, and a free-flowing particulate mixture which comprises at least one fat, at least one starch, and at least one sugar which have been mixed and heated, said at least one starch having a degree of gelatinization of less than about 30%; and

lactobacillus encapsulated by the matrix.

* * * * *

Anexo 2

US006086927A

United States Patent [19]

Frielich et al.

[11] **Patent Number: 6,086,927**

[45] **Date of Patent: Jul. 11, 2000**

[54] **PROCESS FOR PREPARING CALCIUM ENRICHED FOOD PRODUCTS AND THE PRODUCTS THEREFROM**

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[52] **U.S. Cl.** **426/74**; 426/599; 426/615; 426/616

[58] **Field of Search** 426/74, 599, 615, 426/616

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[57] **ABSTRACT**

A process for preparing a calcium enriched food product by:

A) forming a mixture of calcium hydroxide and water or a fruit juice;

B) mixing phosphorus-based acid with the mixture, the phosphorus-based acid being in an amount sufficient to substantially neutralize the calcium hydroxide or to obtain a pH in the range of from about 4.0 to about 6.0;

C) mixing calcium lactate with the mixture from step B) until the calcium lactate is dispersed into the mixture; and

D) mixing calcium phosphate with the mixture from step C) until the calcium phosphate is dispersed into the mixture. Beverage material, especially fruit juice is mixed with the mixture from step D) so as to form a calcium enriched food product. A process is further provided wherein the components are directly added into fruit juice, particularly not from concentrate fruit juice. Calcium enriched orange juice made by the process can exhibit taste and color characteristics similar to non-calcium enriched orange juice.

41 Claims, No Drawings

PROCESS FOR PREPARING CALCIUM ENRICHED FOOD PRODUCTS AND THE PRODUCTS THEREFROM

BACKGROUND OF THE INVENTION

A. Field of the Invention

The present invention relates to a process for preparing calcium enriched food products, particularly beverages, and the products that can be prepared by the process.

B. Description of the Related Art

Calcium is essential to the well being of humans and is continuously utilized by the body. In particular, calcium is used in the body to provide rigidity to the skeletal framework, as a catalyst for the conversion of prothrombin to thrombin, a compound necessary for blood clotting, to increase cell membrane permeability, to activate a number of enzymes including lipase and adenosine triphosphatase, and to act as a component in the mechanisms of neural transmission and muscular contraction.

Calcium can be obtained from a variety of dietary sources. Primary sources of calcium are dairy products, in particular milk, which account for 75% of the daily calcium intake while foods other than dairy products generally contribute less than 200 mg of calcium daily. However, beginning in young adulthood and continuing through later life, the general population may not consume milk in sufficient quantities to obtain the recommended dietary levels of calcium.

Calcium deficiencies have been noted as a major health problem, particularly for women. Osteoporosis, an accelerated bone loss, can occur when the body is deficient in calcium. During a period of calcium deficiency, calcium that is needed for various body functions can be retrieved from bones and thereby prevent bone remodeling. Premenopausal adult women require about 1,000 milligrams of calcium per day (based on recommendations by the 1984 NIH Consensus Development Panel on Osteoporosis). Younger women, particularly pregnant and lactating women and postmenstrual women may require more. Further, adequate calcium intake before age 35 may lessen the effect of osteoporosis in later life.

To provide additional sources of calcium, a variety of calcium supplements have been developed. Calcium carbonate, calcium lactate, and calcium gluconate are commonly used. Calcium carbonate has 40 percent calcium and is generally available in tablet form. Calcium lactate has 13 percent calcium and calcium gluconate has 9 percent calcium. The problems encountered with tablets are that they may be difficult to swallow, or if chewable, can leave an unpleasant "chalky" taste in the mouth.

To meet this situation, the art has developed a number of calcium enriched beverages. Such beverages have ranged from enriched milk products to a sweetener supplement containing soluble calcium, citric acid, malic acid and sugar, as described in U.S. Pat. No. 5,468,506. Fruit juices have received particular attention as a vehicle for providing additional calcium to the diet. For instance, U.S. Pat. No. 4,722,847 describes fruit juice beverages and concentrates by forming a premix solution containing highly soluble calcium citrate and malate species which is then combined with concentrated fruit juice, plus other fruit juice materials. The method is said to provide beverages and concentrates which contain substantial levels of solubilized calcium without generating cooked/browned off-flavors and without including undesirable species such as chloride ions.

U.S. Pat. No. 4,871,554 describes a calcium fortified beverage that contains water, a concentrated fruit juice and a solubilized calcium component derived from a salt blend wherein 50–80% by weight of total calcium is tribasic calcium phosphate and 20–50% by weight of total calcium is calcium lactate. A number of other documents describing calcium enriched beverages is described in the background of the foregoing patents.

The large number of documents describing calcium enriched beverages reflects the difficulty in providing an acceptable product. Calcium compounds can be difficult to dissolve into fruit juice beverages unless expensive mixing equipment is used and can impart undesirable color and/or unpleasant taste to the beverage. Furthermore, certain of the calcium compounds used to enrich the beverages are relatively expensive and significantly increase the cost of the calcium enriched product.

SUMMARY OF THE INVENTION

In one aspect, the present invention provides a process for preparing a calcium enriched food product comprising:

A) forming a mixture of calcium hydroxide and water;

B) mixing a phosphorus-based acid with the mixture, said phosphorus-based acid being in an amount sufficient to substantially neutralize the calcium hydroxide;

C) mixing calcium lactate with the mixture from step B) until the calcium lactate is dispersed into the mixture;

D) mixing calcium phosphate with the mixture from step C) until the calcium phosphate is dispersed into the mixture; and

E) mixing the mixture from step D) with fruit juice so as to form a calcium enriched food product.

In a further aspect, the present invention provides a process for preparing a calcium enriched food product comprising:

A) forming a mixture of calcium hydroxide and fruit juice;

B) mixing a phosphorus-based acid with the mixture, said phosphorus-based acid being in an amount sufficient to substantially neutralize the calcium hydroxide;

C) mixing calcium lactate with the mixture from step B) until the calcium lactate is dispersed into the mixture; and

D) mixing calcium phosphate with the mixture from step C) until the calcium phosphate is dispersed into the mixture so as to form a calcium enriched food product.

The present invention also provides a calcium enriched food product that is made by the processes.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

As noted above, one aspect of the present invention relates to a process for preparing a calcium enriched food product wherein a mixture of calcium hydroxide and water is first formed. The calcium hydroxide is of food grade purity (FCC certified (compliant)), is generally at least about 95% by weight, preferably from about 96.5 to about 98% by weight pure, and is typically in the form of powder having a particle size such that about 96.5% by weight passes through a 325 mesh sieve. The calcium hydroxide is available from a number of sources, such as Mississippi Lime Management Company distributed by Bell Chem. Corp. of Longwood, Fla., under the designation hydrated lime.

To form the calcium hydroxide and water mixture, the calcium hydroxide is added to water in a first container. As

can be understood by those knowledgeable in the art, the calcium hydroxide can be added as calcium oxide which converts to calcium hydroxide in the presence of water. The container is typically in the form of a stainless steel kettle provided with an agitator. The size of the kettle is preferably selected so as to provide a sufficient volume depending on the size of the batch. For instance, if a 1,000–2,000 gallon batch of final product is to be prepared, the volume of the first container can be about 250 gallons.

The calcium hydroxide is added in an amount that depends on the final product and the level of calcium fortification. For instance, for the production of typical concentrated fruit juices (e.g., orange juice) that are four-fold concentrated (which are reconstituted by combining three volumes of water with one volume of concentrate) and are designed to provide 30% of the recommended daily intake (RDI) for calcium per 8 fl. oz. serving, calcium hydroxide is added to form from about 2.6 to about 3.6% weight to volume of the mixture, preferably from about 3.1 to about 3.3% weight to volume of the mixture. Of course, concentrations outside of these ranges can be used if it is convenient to handle larger volumes of the mixture or a single strength product is to be prepared where substantially all of the water used to reconstitute the concentrate is derived from the calcium-containing mixture. During mixing of the calcium hydroxide in the kettle, as well as subsequent mixing steps, the temperature of the contents of the kettle is from about 25 to about 35° C. Mixing of the calcium hydroxide is conducted for a time suitable to completely disperse the calcium hydroxide in the water. This usually involves mixing for from about 5 to about 15 minutes, but is dependent on the mixing equipment utilized.

To the calcium hydroxide and water mixture is added a phosphorus-based acid in an amount sufficient to substantially neutralize the calcium hydroxide. The phosphorus-based acid can be phosphorous acid, ortho-phosphoric acid (commonly referred to as phosphoric acid), meta-phosphoric acid, polyphosphoric acid, pyrophosphoric acid or material which can provide such acids, such as phosphoric anhydride. Mixtures of the phosphorus-based acid may also be used. The selected phosphorus-based acid must be food grade. The preferred phosphorus-based acid is phosphoric acid, particularly phosphoric acid that has a purity of at least about 85%, preferably from about 84.5% to about 85.5% by weight. Phosphorus-based acids are commercially available and an especially suitable phosphoric acid is commercially available from Wilson Co. of Aurora, N.C. distributed by Bell Chem. Corp. of Longwood, Fla., under the designation Albrite 85% Phosphoric Acid Food Grade. The phosphorus-based acid is typically added to the mixture of calcium hydroxide and water in the form of an aqueous solution at a concentration that is again selected depending on the final product and the desired % RDI for calcium per serving. For a typical four-fold concentrated fruit juice, the concentration of phosphorus-based acid in the aqueous solution is from about 3.1 to about 4.3% weight to volume, especially when the phosphorus-based acid is phosphoric acid (which again can vary outside of this range) and is mixed in the first container so as to obtain a mixture having a pH of from about 6 to about 7, preferably about 6.8. Mixing of the phosphorus-based acid with the calcium hydroxide is typically conducted for from about 8 to about 15 minutes, optimally about 12 minutes.

Although it is preferred from the standpoints of the flavor of the final product and the balance of natural fruit acids that the phosphorus-based acid be exclusively used to neutralize the calcium hydroxide, a portion of the phosphorus-based acid (generally less than about 25% by weight of the total acid) can be replaced with a food grade mineral acid (e.g., sulfuric acid) to obtain a pH within the aforementioned range of from about 6 to about 7.

Food grade calcium lactate is next added to the first container and mixing is conducted so that the calcium lactate is thoroughly dispersed. The calcium lactate is added in the form of granules or preferably powder from the standpoint of ease of dispersion and mixing is conducted for from about 3 to about 7 minutes so that thorough dispersion is achieved. The calcium lactate is preferably in hydrated form and is commercially available from Purac Biochemical of Holland distributed by Bell Chem. Corp. of Longwood, Fla., under the designation Puracal PP. The amount of calcium lactate added to the mixture is again selected depending on the final product and the desired % RDI for calcium per serving, but a four-fold concentrated fruit juice that provides 30% RDI for calcium per 8 fl. oz. serving, the calcium lactate is added to the first container in an amount such that the concentration is from about 4.0 to about 5.6% weight to volume (which can again vary outside of this range depending on factors such as amount of mixture which is desired and the type of final product). Although not preferred, a small portion of the calcium lactate (e.g., less than about 20% by weight of the calcium lactate) can be replaced with calcium sulfate. However, the amount of calcium sulfate should be selected so as to not substantially adversely affect the flavor of the final product.

Food grade calcium phosphate is then added to the first container and mixing is conducted so that the calcium phosphate is thoroughly dispersed. The calcium phosphate can be mono-calcium phosphate, di-calcium phosphate, tri-calcium phosphate or mixtures thereof. Tri-calcium phosphate is preferred in view of the flavor of the final product. The calcium phosphate is generally added in the form of powder and mixing is conducted for from about 8 to about 12 minutes so that thorough dispersion is achieved. The calcium phosphate is commercially available from various sources. For instance, tri-calcium phosphate is commercially available from Gadot Biochemical of Israel distributed by Bell Chem. Corp. of Longwood, Fla. under the designation Tri Calcium Phosphate FCC and has a particle size such that a minimum of 95% by weight passes through a 325 mesh sieve.

Upon completion of the addition and mixing of the calcium phosphate, the mixture comprises from about 34 to about 48% by weight of total calcium derived from calcium hydroxide, from about 12 to about 18% by weight of total calcium derived from calcium lactate and from about 40 to about 48% by weight of total calcium derived from calcium phosphate. Preferably, the mixture comprises from about 37 to about 43% by weight of total calcium derived from calcium hydroxide, from about 14 to about 16% by weight of total calcium derived from calcium lactate and from about 43 to about 47% by weight of total calcium derived from calcium phosphate. In an especially preferred embodiment, the mixture comprises about 40% by weight of total calcium derived from calcium hydroxide, about 15% by weight of total calcium derived from calcium lactate and about 45% by weight of total calcium derived from calcium phosphate.

Upon completion of the calcium phosphate mixing step, the contents of the first container are added to a beverage material that can be naturally or artificially flavored and/or sweetened that is maintained in a separate second container. A preferred beverage material is fruit juice. While any fruit juice can be used, especially advantageous are citrus fruit juices, particularly orange juice and/or grapefruit juice. The

fruit juice can be in concentrated form such as 30 to 65 brix as measured by a refractometer in which values have been corrected for acidity. Concentration of the fruit juices can be conducted in any technique known to those of ordinary skill in the art, such as a falling film evaporator. An especially preferred concentrate is concentrated orange juice for manufacturing as defined in 21 C.F.R. §146.153 (the contents of title 21 of the Code of Federal Regulations being incorporated by reference). Thus, for instance, the concentrated orange juice for manufacturing can be thawed frozen orange juice which has been five-fold concentrated so that when the calcium-containing mixture is added to the concentrate (with or without subsequently added rinse water), a four-fold concentrate is obtained which can be packaged, frozen and ultimately sold and which can be reconstituted by combining three volumes of water with one volume of concentrate.

The second container is generally a stainless steel tank having a volume that is dependent on the desired batch size. The tank is provided with an agitator and can be maintained at a temperature of from about 2 to about 10° C. The calcium-containing mixture is mixed with the fruit juice for from about 10 to about 15 minutes so that complete dispersion is achieved. If desired, juice oil and essences, such as orange oil and essences or grapefruit oil and essences can be added to the second container to improve the characteristics of the final product before or after the addition of the calcium-containing mixture.

In order to ensure that all of the calcium-containing mixture is transferred from the first container to the second container holding the fruit juice, rinse water is usually sprayed into the first container and the rinse water is then added to the fruit juice mixture in the second container and mixed therewith. Typically, the amount of water added to the first container is from about 30 to about 50% of the volume of the contents of the first container. The calcium concentration of the mixture can then be determined. If additional calcium is warranted, a thoroughly mixed water and calcium lactate mixture can be added. Additional water may be directly added to the second container so that a calcium enriched fruit juice beverage is formed. The calcium enriched fruit juice beverage can then be packaged for transportation and sale or can be transferred to a bulk tank for shipment and later packaging. Thus, for example, four-fold concentrated orange juice made by the foregoing process can be packed into containers and blast frozen to produce calcium enriched frozen concentrated orange juice that the end user will reconstitute with three containers of water to produce a calcium enriched single strength product that can provide 30% RDI for calcium per 8 fl. oz. serving. Alternatively, the calcium enriched concentrate can be reconstituted with water and packed into containers to provide a calcium enriched ready-to-serve product that can similarly provide 30% RDI for calcium per 8 fl. oz. serving.

In a further embodiment of the invention, a mixture of fruit juice and calcium hydroxide is initially formed by mixing calcium hydroxide (or calcium oxide which forms calcium hydroxide) with fruit juice rather than water. The fruit juice can be concentrated, such as a concentrate of from about 15 to about 40 brix, but is preferably not from concentrate. Especially preferred is not from concentrate orange juice or grapefruit juice. The process is conducted similar to the process described above with the phosphoric acid, calcium lactate and the calcium phosphate being sequentially added to the mixture of fruit juice and calcium hydroxide. However, upon completion of the phosphoric acid addition, the pH of the mixture can be approximately the pH of the original juice. Thus, for instance, where the fruit juice is orange juice, the pH upon completion of mixing of the phosphoric acid is from about 4.0 to about 5.0, especially about 4.2. On the other hand, where the fruit juice is grapefruit juice, the amount of phosphoric acid is reduce so that the pH in the container is from about 5.0 to about 6.0 so as to yield a more preferred product based on taste.

This embodiment of the invention can provide a high quality, fresh-tasting, not from concentrate juice which is calcium enriched without addition of water. To ensure that all the material is included and uniformly dispersed within the product, a first larger container can essentially be filled with the not from concentrate juice and a portion of the juice can be transferred to a smaller second container. The amount of juice transferred to the second container is selected so that the calcium hydroxide or calcium oxide, phosphoric acid, calcium lactate and calcium phosphate can be sufficiently dispersed in the juice to obtain the desired level of calcium for the entire volume of the first container when the juice in the second container is transferred back to the first container. Thus, for a first container holding 1600 gallons of not from concentrate juice, from about 150 to about 200 gallons can be transferred to a 250 gallon second container equipped with an agitator and the stated materials are added thereto in sufficient quantities such that when the juice is transferred back to the first container, the proper level of calcium enrichment can be attained.

After the juice is transferred back to the first container, the second container can then be rinsed with an additional quantity of juice from the first container (typically from about 30 to about 80% of the volume of the second container) to ensure that all the calcium derived from the described compounds is included within in the juice and to establish an essentially uniform concentration of calcium in the juice in the second container and the lines connecting the first and second containers.

As will be understood, other arrangement can also be used. For instance, the stated materials can be added to a selected quantity of juice in a large container and thereafter additional quantity of juice can be added to the container with mixing so that the materials are fully dispersed in the juice.

By following the teachings of the present invention, a calcium enriched food product can be prepared that is similar in characteristics to the product without calcium enrichment. For example, where calcium enriched orange juice is prepared by the present invention, the orange juice can have a calcium content of from about 1275 ppm to about 1700 ppm and yet have taste, color and aroma characteristics that are very similar to orange juice which has not been calcium enriched. Thus, for example, the calcium enriched juice can provide a substantial source of calcium (e.g., from about 10 to about 40% RDI for calcium per 8 fl. oz. serving) without a "chalky" taste that often accompanied known calcium enriched beverages. Furthermore, the product of the present invention can substantially maintain the calcium in dispersed form. These substantial advantages can be attained without specialized process equipment, such as high shear mixers, and without using more expensive sources of calcium such as calcium citrate or calcium gluconate or more expensive acids, such as citric acid.

The following Examples include lab scale protocols and illustrate various aspects of the present invention. In the Examples, the calcium hydroxide, phosphoric acid, calcium lactate and calcium triphosphate used are obtained from the sources identified above. It is to be understood that the present invention is defined by the appended claims and not the specific details of the Examples.

EXAMPLE 1

Into a 250 ml beaker was added 125 ml of deionized water. A stirring bar was added and the beaker was placed on a stirring plate so that stirring is continuously conducted. Calcium hydroxide in an amount of 4.01 gr was added to the beaker and stirring continued for 10 minutes. Phosphoric acid (85%) was next added to the beaker in an amount of 4.81 gr and stirring was continued for 15 minutes. The pH of the mixture was checked and, if necessary, additional acid was added to obtain a pH in the range of 6 to 7. Calcium lactate was then added to the beaker in an amount of 6.24 gr and stirring continued for 5 minutes. Tri-calcium phosphate was added to the beaker in an amount of 7.16 gr and stirring continued for 5 minutes.

In a separate container is placed 642.08 ml of an orange juice concentrate and pulp blend at 60 Brix which contains added cold press oil and essence. The mixture in the 250 ml beaker is added to the container holding the orange juice concentrate. The beaker is rinsed with 50 ml of deionized water and the rinse water is added to the container holding the orange juice concentrate and the contents are mixed well.

The calcium enriched orange juice was analyzed on the basis of Brix (percentage of soluble sugar solids) using a refractometer; Ratio (the relationship between Brix and acid content measured in % by weight as citric acid that is an indication of the maturity of the fruit); Color (measured by a citrus colorimeter as approved by the U.S. Dept. of Agriculture and the State of Florida); Oil (as determined by the Scott Oil Method described in the U.S. Dept. of Agriculture Handbook, the contents of which are incorporated by reference); COD (the Chemical Oxygen Demand which is a measure of amount of aromatic compounds); and Calcium (determined by the wet chemistry method using titration by ethylene diamine tetraacetic acid). The technique for determining the concentration of calcium is performed on samples of the juice concentrate before (blank) and after calcium addition and is as follows:

The juice sample is reconstituted to 12.2° Brix and 10 ml is pipetted into a centrifuge tube and 1 ml of IN hydrochloric acid is added to the tube. The tube is centrifuged for 10 minutes at 1500 rpm. From the supernatant is pipetted 1 ml which is placed into an Erlenmeyer flask to which is further added 100 ml of distilled water. 2 ml of a 45% potassium hydroxide is pipetted with caution into the flask and one scoop of hydroxy naphthol blue indicator is then added. A stirrer bar is placed in the flask and the flask is placed on a stirrer plate in front of a white background and moderate stirring is conducted. Using a solution of 0.01M ethylene diamine tetraacetic acid (EDTA), the sample is titrated from a wine red to persistent (10 seconds) sharp blue end point. The amount of EDTA solution is recorded and the following calculation performed:

$$Ca^{++}(ppm) = \frac{(ml\ EDTA\ sample - ml\ EDTA\ blank) \times 0.4\ mg/ml\ Ca \times 1.1}{.001\ liters}$$

The foregoing characteristics were compared with those obtained from orange juice made from the same concentrate, but without calcium enrichment (the blank) and the results are provided in the following Table 1.

TABLE 1

PRODUCT	BRIX	RATIO	COLOR	OIL	COD	Ca
Ca Enriched Orange Juice	12.2	16.97	35.5	0.011%	193	1355.6 ppm
Without Ca Enrichment	11.9	18.27	35.3	0.009%	190	211.2 ppm

When orange juice made by this general process was subjected to a taste panel of 45 individuals, 16 individuals preferred the calcium enriched orange juice, 16 individuals preferred the orange juice without calcium enrichment and 13 individuals had no preference.

EXAMPLE 2

The process of Example 1 was repeated with a different source of frozen orange juice concentrate and the same analysis and comparison with the orange juice without calcium enrichment and the results are set forth in Table 2. The Table also includes the percentage of bottom pulp (BP) which is conducted in accordance with a U.S. Dept. of Agriculture test to determine the amount of insoluble material that does not float.

TABLE 2

PRODUCT	BRIX	RATIO	COLOR	OIL	BP	COD	Ca
Ca Enriched Orange Juice	~12.2	19.3	35.5	0.015%	10%	225	1298 ppm
Without Ca Enrichment	~12.2	18.28	35.5	0.017%	12%	219	—

When orange juice made by this general process was subjected to a taste panel of 47 individuals, 20 individuals preferred the calcium enriched orange juice, 13 individuals preferred the orange juice without calcium enrichment and 14 individuals had no preference.

EXAMPLE 3

The process of Example 1 was repeated with thawed reconstituted frozen concentrate orange juice and the same analysis was conducted. A commercially available calcium enriched orange juice was then analyzed and the results are set forth in Table 3.

TABLE 3

PRODUCT	BRIX	RATIO	COLOR	OIL	COD	Ca
Ca Enriched Orange Juice	12.34	17.38	36.2	0.019%	353	1355.2 ppm
Commercial Ca Enriched	12.81	22.47	35.4	0.010%	—	1113.2 ppm

When orange juice made by this general process was subjected to a taste panel of 60 individuals, 23 individuals preferred the calcium enriched orange juice of the present invention, 22 individuals preferred the commercially available calcium enriched orange juice and 15 individuals had no preference.

EXAMPLE 4

The process of Example 1 was repeated with thawed reconstituted frozen concentrate orange juice and the same analysis was conducted. A commercially available calcium

enriched orange juice was similarly analyzed and the results are set forth in Table 4.

TABLE 4

PRODUCT	BRIX	RATIO	COLOR	OIL	BP	TP	COD	Ca
Commercial Ca Enriched	12.71	22.29	35.5	0.014%	10%	1.7	—	1307 ppm
Inventive Ca Enriched	12.2	19.3	35.5	0.015%	10%	—	225	1298 ppm

When orange juice made by this general process was subjected to a taste panel of 48 individuals, 18 individuals preferred the calcium enriched orange juice of the present invention, 15 individuals preferred the commercially available calcium enriched orange juice and 15 individuals had no preference.

Examples 5–8 illustrate large scale techniques illustrating various embodiments of the present invention. Example 5 illustrates the preparation of a calcium enriched frozen orange juice concentrate, Examples 6 and 7 illustrate a not-from-concentrate calcium enriched orange juice beverage and Example 8 illustrates a not-from-concentrate calcium enriched grapefruit juice beverage. In these Examples, the calcium hydroxide, phosphoric acid, tri-calcium phosphate and calcium lactate were obtained from Bell Chem. Corp. of Longwood, Fla.

EXAMPLE 5

In a 250 gallon stainless steel kettle equipped with an agitator, approximately 180 gal of water and 53.54 lbs of calcium hydroxide are added and mixed for at least 10 minutes. To the kettle is then added 64.22 lbs (approx. 4.5 gal) of phosphoric acid and the contents are stirred for approximately 15 minutes until the pH is between 6.00 and 7.00. Calcium lactate in an amount of 83.32 lbs is added to the kettle and mixed for approximately 5 minutes. Tri-calcium phosphate is next added to the kettle in an amount of 95.60 lbs and mixed for at least 10 minutes to obtain complete mixing. A tri-blender can be used to ensure complete mixing.

The resulting slurry is pumped directly to a 1600 gallon stainless steel tank provided with an agitator and containing approximately 927 gallons of orange juice that has been concentrated so as to provide an approximately 7:1 by volume reconstitution ratio. The orange juice concentrate is at a temperature of approximately 2° C. and can contain orange oil and essence complying with Federal Regulations. After the slurry is pumped to the tank containing the orange juice concentrate, approximately 100 gallons of water is added to the kettle, mixed and pumped to the tank containing the mixture of prepared slurry and orange juice concentrate to insure all the calcium is added to the tank. The calcium content is then checked and, if calcium concentration is low, a calcium lactate mixture with water, can be added to the calcium enriched orange juice concentrate. Water is then added to obtain the desired brix within the range of 42.5 to 42.9 as determined by refractometer. In addition, the product has a color on the order of 35.5 and a brix:acid ratio in the range of 14.0: 1 to 20.5: 1. The final product of approximately 1600 gallons is agitated a minimum of 15 minutes before filling containers at a temperature not greater than about 4° C. then flash freezing the containers.

Upon 3:1 volume reconstitution of the orange juice concentrate with water, the orange juice exhibits a brix of approximately 12.2 and provides approximately 30% RDI for calcium per 8 fl. oz. serving (approximately 1270 ppm calcium).

EXAMPLE 6

A 1600 gallon stainless steel tank provided with an agitator is essentially filled with not from concentrate orange juice. Approximately 200 gallons of the not from concentrate orange juice is transferred into a 250 gallon stainless steel kettle equipped with an agitator at a temperature of approximately 4° C. To the kettle is added 13.39 lbs of calcium hydroxide and the contents are mixed for at least 5 minutes. Phosphoric acid in an amount of 25.7 lbs is added to the kettle and the contents mixed for at least 10 minutes until the pH is between 4.0 and 5.0. Calcium lactate in an amount of 20.83 lbs is added to the kettle and the contents mixed for approximately 5 minutes. Calcium tri-phosphate in an amount of 23.90 lbs is next added to the kettle and mixed for about 10 minutes.

The contents of the kettle are pumped back to the 1600 gallon stainless steel tank and mixed for at least 5 minutes. Into the 250 gallon kettle is introduced approximately 200 more gallons of the not-from-concentrate orange juice from the 1600 gallon stainless steel tank, the contents of the kettle are stirred and returned to the 1600 gallon stainless steel tank to ensure that the calcium is homogeneously distributed in the juice in the 1600 gallon stainless steel tank. If desired, orange oil and essence can be added if desired in amounts complying with Federal Regulations.

The calcium enriched orange juice can be filled into retail containers or bulk shipped for later packaging. The calcium enriched orange juice exhibits a brix in the range of from 11.2 to 13.0, a brix:acid ratio of 16.5:1 to 19.5:1 and a color on the order of 37. The calcium enriched orange juice can provide 30% RDI for calcium per 8 fl. oz. serving (approximately 1270 ppm).

EXAMPLE 7

A 1600 gallon stainless steel tank provided with an agitator is essentially filled with not from concentrate orange juice. Approximately 200 gallons of the not from concentrate orange juice is transferred into a 250 gallon stainless steel kettle equipped with an agitator at a temperature of approximately 4° C. To the kettle is added 14.69 lbs of calcium hydroxide and the contents of the kettle are mixed for at least 5 minutes. Phosphoric acid in an amount of 28.2 lbs is added to the kettle and the contents mixed for at least 10 minutes until the pH is between 4.0 and 5.0. Calcium lactate in an amount of 22.83. lbs is then added to the kettle and mixed well for approximately 5 minutes. Tri-calcium phosphate in an amount of 26.17 lbs is next added to the kettle and mixed for about 10 minutes.

The contents of the kettle are pumped back to the 1600 gallon stainless steel tank and agitated for at least 5 minutes. Into the kettle is introduced approximately 200 more gallons of the not-from-concentrate orange juice from the 1600 gallon stainless steel tank, the contents of the kettle are stirred and returned to the 1600 gallon stainless steel tank to ensure that the calcium is homogeneously distributed in the

juice in the 1600 gallon stainless steel tank. If desired, oil and ascorbic acid can be added in amounts complying with Federal Regulations.

The calcium enriched orange juice can be filled into retail containers or bulk shipped for later packaging. The calcium enriched orange juice exhibits a brix in the range of from 11.3 to 13.0, a brix:acid ratio of 15.0:1 to 20.5:1 and a color on the order of 35. The calcium enriched orange juice can provide 35% RDI for calcium per 8 fl. oz. serving (approximately 1479 ppm).

EXAMPLE 8

A 1600 gallon stainless steel tank provided with an agitator is essentially filled with not from concentrate grapefruit juice. Approximately 200 gallons of the not from concentrate grapefruit juice is transferred into a 250 gallon stainless steel kettle equipped with an agitator at a temperature of approximately 4° C. To the kettle is added 13.40 lbs of calcium hydroxide and the contents of the kettle are mixed for at least 5 minutes. Phosphoric acid in an amount of 8.0 lbs is added to the kettle and the contents mixed for at least 10 minutes until the pH is between 5.0 and 6.0. Calcium lactate in an amount of 20.85 lbs is then added to the kettle and the contents are mixed well for approximately 5 minutes. Tri-calcium phosphate in an amount of 23.92 lbs is next added to the kettle and mixed for about 10 minutes.

The contents of the kettle are pumped back to the 1600 gallon stainless steel tank and agitated for at least 5 minutes. Into the kettle is introduced approximately 200 more gallons of the not-from concentrate grapefruit juice from the 1600 gallon stainless steel tank, the contents of the kettle are stirred and returned to the 1600 gallon stainless steel tank to ensure that the calcium is homogeneously distributed in the juice in the 1600 gallon stainless steel tank.

The calcium enriched grapefruit juice can be filled into retail containers or bulk shipped for later packaging. The calcium enriched orange juice exhibits a brix in the range of from 9.4to 12.4, a brix:acid ratio of 10.0:1 to 15.0:1 and a color on the order of 18. The calcium enriched orange juice can provide 30% RDI for calcium per 8 oz. serving (approximately 1270 ppm).

While the invention has been described in detail with reference to preferred embodiments thereof, it will be apparent to one skilled in the art that various changes can be made, and equivalents employed, without departing from the scope of the invention.

What is claimed is:

1. A process for preparing a calcium enriched food product comprising:

A) forming a mixture of calcium hydroxide and water;

B) mixing phosphorus-based acid with the mixture, said phosphorus-based acid being in an amount sufficient to substantially neutralize the calcium hydroxide;

C) mixing calcium lactate with the mixture from step B) until the calcium lactate is dispersed into the mixture;

D) mixing calcium phosphate with the mixture from step C) until the calcium phosphate is dispersed into the mixture wherein from about 34 to about 48% by weight of total calcium is derived from calcium hydroxide, from about 12 to about 18% by weight of total calcium is derived from calcium lactate and from about 40 to about 48% by weight of total calcium is derived from calcium phosphate; and

E) mixing the mixture from step D) with beverage material so as to form a calcium enriched food product.

2. The process of claim 1 wherein the calcium hydroxide is mixed with water in an amount of from about 2.6 to about 3.6% weight to volume of the mixture.

3. The process of claim 1 wherein the calcium hydroxide is mixed with water in an amount of from about 3.1 to about 3.3% weight to volume of the mixture.

4. The process of claim 1 wherein the phosphorus-based acid is added in an amount of from about 3.1 to about 4.3% weight to volume.

5. The process of claim 4 wherein the phosphorus-based acid is added in the form of an aqueous solution.

6. The process of claim 1 wherein upon completion of step B), the mixture has a pH of from about 6 to about 7.

7. The process of claim 1 wherein calcium lactate is mixed in the form of powder or granules.

8. The process of claim 1 wherein the calcium phosphate is tri-calcium phosphate.

9. The process of claim 1 wherein upon the completion of step D), the mixture comprises from about 37 to about 43% by weight of total calcium is derived from calcium hydroxide, from about 14 to about 16% by weight of total calcium is derived from calcium lactate and from about 43 to about 47% by weight of total calcium is derived from calcium phosphate.

10. The process of claim 1 wherein upon the completion of step D), the mixture comprises about 40% by weight of total calcium derived from calcium hydroxide, about 15% by weight of total calcium derived from calcium lactate and about 45% by weight of total calcium derived from calcium phosphate.

11. The calcium enriched food product made by the process of claim 10.

12. The process of claim 1 wherein the beverage material is concentrated fruit juice.

13. The process of claim 12 wherein steps A) through E) are conducted in a first container and the mixture from step E) is added to concentrated fruit juice maintained in a second container.

14. The process of claim 13 wherein after the mixture from step E) is added to concentrated fruit juice maintained in the second container, water is added to the first container so as to mix with residual material therein and the mixture is added to the second container.

15. The process of claim 12 wherein after step E), water is added to the calcium enriched food product so as to form a calcium enriched beverage.

16. The process of claim 15 wherein the amount of water is sufficient to reconstitute the fruit juice to a Brix value substantially the same as not from concentrate fruit juice.

17. The process of claim 15 wherein the fruit juice is orange juice, grapefruit juice or mixtures thereof.

18. The calcium enriched food product made by the process of claim 17.

19. The calcium enriched food product of claim 18 wherein the beverage has a calcium content so as to provide from about 10 to about 40% of the RDI for calcium per 8 oz. serving.

20. The calcium enriched food product made by the process of claim 15.

21. The process of claim 1 wherein the temperature of steps A) through D) is from about 20 to about 35° C.

22. The process of claim 1 wherein the temperature of step E) is from about 2 to about 10° C.

23. The process of claim 1 wherein the phosphorus-based acid is phosphoric acid.

24. The process of claim 1 wherein the fruit juice is orange juice, grapefruit juice or a mixture thereof.

25. The calcium enriched food product made by the process of claim 1.

26. A process for preparing a calcium enriched food product comprising:

A) forming a mixture of calcium hydroxide and fruit juice;

B) mixing phosphorus-based acid with the mixture, said phosphorus-based acid being in an amount to obtain a pH in the range of from about 4.0 to about 6.0;

C) mixing calcium lactate with the mixture from step B) until the calcium lactate is dispersed into the mixture; and

D) mixing calcium phosphate with the mixture from step C) until the calcium phosphate is dispersed into the mixture so as to form a calcium enriched food product wherein from about 34 to about 48% by weight of total calcium is derived from calcium hydroxide, from about 12 to about 18% by weight of total calcium is derived from calcium lactate and from about 40 to about 48% by weight of total calcium is derived from calcium phosphate.

27. The process of claim 26 wherein the fruit juice is orange juice.

28. The process of claim 27 wherein the orange juice is not-from-concentrate orange juice.

29. The process of claim 26 wherein the calcium hydroxide is mixed with the fruit juice so as to be present in an amount of from about 0.6 to about 1.0% weight to volume of the mixture.

30. The process of claim 26 wherein the calcium hydroxide is mixed with the fruit juice so as to be present in an amount of from about 0.8 to about 0.9% weight to volume of the mixture.

31. The process of claim 26 wherein the fruit juice is orange juice and the phosphorus-based acid is added in an amount to obtain a pH in the range of from about 4.0 to about 5.0.

32. The process of claim 31 Wherein upon completion of step B), the mixture has a pH of about 4.2.

33. The process of claim 26 wherein the fruit juice is grapefruit juice and the phosphorus-based acid is added in an amount to obtain a pH in the range of from about 5.0 to about 6.0.

34. The process of claim 26 wherein calcium lactate is mixed in the form of powder or granules.

35. The process of claim 26 wherein the phosphorus-based acid is phosphoric acid.

36. The process of claim 67 wherein the calcium phosphate is tri-calcium phosphate.

37. The process of claim 26 wherein upon the completion of step D), the mixture comprises from about 37 to about 43% by weight of total calcium is derived from calcium hydroxide, from about 14 to about 16% by weight of total calcium is derived from calcium lactate and from about 43 to about 47% by weight of total calcium is derived from calcium phosphate.

38. The process of claim 26 wherein upon the completion of step D), the mixture comprises about 40% by weight of total calcium derived from calcium hydroxide, about 15% by weight of total calcium derived from calcium lactate and about 45% by weight of total calcium derived from calcium phosphate.

39. The calcium enriched food product of claim 38.

40. The calcium enriched food product made by the process of claim 26.

41. The calcium enriched food product of claim 40 wherein the beverage has a calcium content so as to provide from about 10 to about 40% of the RDI for calcium per 8 oz. serving.

* * * * *

Industria y Comercio
SUPERINTENDENCIA

2020
Bogotá, D.C.,

Doctora:
Dilia María Rodríguez D'Aleman
Apoderada

Oficio N° 092061

ASUNTO:

Radicación PCT Nº	08-83008
Trámite	011
Evento	379
Actuación	440
Folios	008

Con fundamento en lo previsto por el artículo 43 de la Decisión 486 de la Comisión de la Comunidad Andina, se le comunica que la oposición presentada por el doctor Tito Noe Parra Murillo, en su calidad de apoderado de Lafrancol S.A., reúne los requisitos formales exigidos por la ley al momento de su presentación.

En cumplimiento de lo dispuesto en la citada norma, se le concede el término de sesenta (60) días hábiles siguientes a la fecha de notificación del presente oficio, para que haga valer sus argumentaciones y presente pruebas.

Notifíquese el presente oficio conforme a lo dispuesto en el numeral 5.2, literal e), del capítulo quinto del título primero de la Circular Única Nº 10 de 2001.

ALIX CARMENZA CESPEDES DE VERGEL
Directora de Nuevas Creaciones (C).

El anterior oficio fue notificado por fijación en lista de fecha, 23 JUL. 2010.

202021

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Industria y Comercio
SUPERINTENDENCIA

2020
Bogotá, D.C.,

Doctora:
Dilia María Rodríguez D'Aleman
Apoderada

Oficio N°: 09207

ASUNTO:

Radicación PCT Nº	08-83008
Trámite	011
Evento	379
Actuación	440
Folios	010

Con fundamento en lo previsto por el artículo 43 de la Decisión 486 de la Comisión de la Comunidad Andina, se le comunica que la oposición presentada por la doctora Cielo Ángela Peña Rodríguez, en su calidad de apoderada de Procaps S.A., reúne los requisitos formales exigidos por la ley al momento de su presentación.

En cumplimiento de lo dispuesto en la citada norma, se le concede el término de sesenta (60) días hábiles siguientes a la fecha de notificación del presente oficio, para que haga valer sus argumentaciones y presente pruebas.

Notifíquese el presente oficio conforme a lo dispuesto en el numeral 5.2, literal e), del capítulo quinto del título primero de la Circular Única Nº 10 de 2001.

ALIX CARMENZA CESPEDES DE VERGEL
Directora de Nuevas Creaciones (C).

El anterior oficio fue notificado por fijación en lista de fecha, 23 JUL. 2010.

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