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SUPERINDUSTRIA Y COMERCIO

Radicacion : 00074264 00000003

Folios: 2

Fecha (AMD): 2003-02-14 09:37:38

Tramite : 002 PATENTES D 1 REGISTRADO/ 538 PETICION

Dependencia: 2020 DIVISION DE NUEVAS CREACIONES

SEÑORES

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO

DIVISIÓN DE NUEVAS CREACIONES

MINISTERIO DE DESARROLLO ECONOMICO

REF: EXPEDIENTE : **00074264**

SOLICITANTE: GENERAL MILLS, INC

FECHA DE LA SOLICITUD: SEPTIEMBRE 29, 2000

TITULO: ENCAPSULADO DE COMPONENTES SENSIBLES EN UNA
MATRIZ PARA OBTENER PARTICULAS DISCRETAS ESTABLES
DURANTE EL ALMACENAMIENTO

ASUNTO: SOLICITUD ESTUDIO DE FONDO

DILIA RODRÍGUEZ D'ALEMAN, en mi condición de apoderada de la Sociedad solicitante, por, el presente escrito atentamente solicito a Usted que habiéndose surtido la publicación de la presente solicitud, en la Gaceta de la Propiedad Industrial N°.520 de fecha Septiembre 27 de 2002 se proceda con el estudio de Fondo.

Para tal fin me permito anexar la tasa correspondiente.

Agradezco continuar con el presente trámite.

Señor Jefe,


DILIA RODRÍGUEZ D'ALEMAN
T.P.No. 20824 del CSJ

P-366

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SUPERINDUSTRIA Y COMERCIO	
Radicacion : 00074264 00000003	Folios: 2
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Dependencia: 2020 DIVISION DE NUEVAS CREACIONES	

NIT : 800176089-2

RECIBO OFICIAL DE CAJA : 03 - 7,985
FECHA : FEBRERO 7 DE 2003

***** CONSIGNACION *****

DEPOSITANTE	TIPO PAGO	BANCO	CUENTA	No. PAGO	FECHA PAGO	Vr. PAGO
CLARKE MODET Y CO. COLOMB	CONSIGNACION	BANCO POPULAR	050-00110-6	9018618	06/02/2003	3,258,000.00

***** CONCEPTO *****

CANT.	RENTISTICO	CONCEPTO	TOTAL CONCEPTO
1	50005-01-01	SOLICITUDES	
		85 EXAMEN DE PATENTABILIDAD DE INVEN	335,000.00
		TOTAL :	\$ 335,000.00

SON: TRESCIENTOS TREINTA Y CINCO MIL PESOS

RESPONSABLE :

RECIBO DE CAJA APLICADO AL EXPEDIENTE No. _____

**SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
DIVISIÓN DE NUEVAS CREACIONES**

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43099

2020/
Bogotá, D.C.,

Doctora
Dilia María Rodríguez
Apoderado

Oficio N°

13459

REFERENCIA	Radicación	00 74264
	Trámite	002
	Evento	001
	Actuación	430
	Folios	021

Como resultado del examen de patentabilidad de la solicitud de patente de invención N° 00 74264, realizado según el artículo 45 de la decisión 486 de la comisión de la Comunidad Andina, se notifica:

Documentos estudiados:

- . Descripción: folios 4-44
- . Ejemplos: folios 44-49
- . Capítulo reivindicatorio estudiado: folios 50-61
- . Se reivindica prioridad, presentada en Estados Unidos el 01.10.99, bajo el número 09/410017

Objeto de la invención

Según las reivindicaciones 1 a 81 de la presente solicitud, se refiere a un método para encapsular o incluir un componente en una matriz, caracterizado porque comprende obtener una masa moldeable, moldear dicha mezcla y secarla. También se refiere a los productos obtenidos mediante dicho procedimiento.

Clasificación internacional de patentes A 61 K 9/26

Claridad de la solicitud

El art. 30 de la decisión 486 contempla que *"Las reivindicaciones definirán la materia que se desea proteger mediante la patente. Deben ser claras y concisas y estar enteramente sustentadas por la descripción"*

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Teniendo en cuenta lo anterior, las reivindicaciones contienen una serie de términos poco claros como:

- "sustancialmente no plastificable no plastificable a temperaturas menores que la temperatura de descomposición del encapsulante", en una situación donde "encapsulante" cubre todo tipo de compuestos posibles que cumplen con esta condición.
- "sustancialmente no destruye dicho encapsulante" depende de términos poco claros como "baja velocidad de cizalla", "baja temperatura", "sin destruir sustancialmente el encapsulante" y "sin gelatinizar o cocinar" no delimitan la materia, ni las condiciones del método reivindicado
- "mezcla moldeada sustancialmente homogénea" no define ni delimita las características del producto obtenido.

Favor hacer las aclaraciones y correcciones del caso, teniendo en cuenta que un método de fabricación se define por sus condiciones específicas de temperatura, tiempo, etc., que delimiten de manera efectiva la materia que se desea proteger.

Determinación del estado de la técnica:

Consultadas las fuentes de información con que cuenta este despacho, se encontraron los siguientes documentos, anteriores a la fecha de presentación de la presente solicitud - 01.10.99-, relacionados con la materia de la presente solicitud:

Nº	Documento	Título	Fecha de publicación
1	WO 9818610	"Incrustación y encapsulado de partículas de liberación controlada"	07.05.98
2	US 4106991	"Composición de granulado de enzimas y proceso para formar granulados de enzimas"	15.08.78
3	US 4242219	"Partículas novedosas de enzimas y su preparación"	30.12.80
4	US 5079012	"Producto de galletería con cubierta estable que contiene aditivos sensibles al calor y corte y su método de preparación"	07.01.92

Evaluación de los requisitos de patentabilidad:

El artículo 14 de la decisión 486 de la comisión de la comunidad andina establece que: *"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"*

En cumplimiento del artículo 14 de la decisión 486 de la comisión de la comunidad andina, se procedió a evaluar los requisitos de novedad, nivel inventivo y aplicación industrial con el fin de establecer la patentabilidad de la invención reivindicada en la solicitud en estudio.

Evaluación de la NOVEDAD

El artículo 16 de la decisión 486 contempla que *"Una invención se considerará nueva cuando no está comprendida en el estado de la técnica"*

La publicación WO 9818610 se relaciona con un método para encapsular un componente en una matriz que comprende:

- obtener una mezcla de un material plastificable de matriz, un plastificante y un encapsulante a baja velocidad de cizalla y baja temperatura
- cortar las piezas, moldearlas y secarlas

La patente US 4106991 describe un proceso para encapsular proteínas que comprende los pasos de.

- mezclar la composición de polvos secos
- añadir un plastificante líquido
- cortar las piezas

La patente US 4242219 publica un proceso para preparar partículas que contienen enzimas que comprende:

- mezclar la enzima en forma seca con una matriz, un plastificante y una cubierta
- cortar, moldear y secar

La patente US 5079012 revela el método de preparación de preparados alimenticios que comprende:

- mezcla de la composición de partes secas
- añadido de un plastificante líquido
- cortado, moldeado y secado

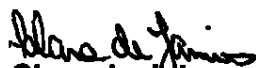
Comparando elemento por elemento, los procedimientos y los productos de las reivindicaciones 1-81 son iguales a las de las anterioridades citadas y pueden afectar la novedad de la presente solicitud.

En conclusión los requisitos de patentabilidad de la presente solicitud se encuentran afectados así:

Nº	Documento	Reivindicaciones afectadas	Requisito que afecta
1	WO 9818610	1-81	Novedad
2	US 4106991	1-81	Novedad
3	US 4242219	1-81	Novedad
4	US 5079012	1-81	Novedad

En cumplimiento del artículo 45 de la Decisión 486 de la Comisión de la Comunidad Andina, se le indica que debe dar respuesta dentro del término de sesenta días contados a partir de la fecha de la notificación de la presente comunicación. En caso de no dar respuesta al presente requerimiento, o si a pesar de la respuesta subsistieran los impedimentos para la concesión, se denegará la patente.

Notifíquese el presente oficio conforme a lo dispuesto en el numeral 5.2, literal e), del capítulo 5 del título primero de la Circular Única N° 10 de 2001


Clara de Jalmes

Jefe de la División de Nuevas Creaciones (E)

El anterior requerimiento fue notificado por fijación en lista, de fecha

22 DIC. 2005

CONCEPTO TÉCNICO Número 1985	EXAMINADOR TÉCNICO Código 202024
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PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : B29C 47/04, B01J 13/04, A01N 25/26	A1	(11) International Publication Number: WO 98/18610
		(43) International Publication Date: 7 May 1998 (07.05.98)
(21) International Application Number: PCT/US97/18984		(81) Designated States: AU, CA, JP, NO, PL, US, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).
(22) International Filing Date: 27 October 1997 (27.10.97)		
(30) Priority Data: 60/029,038 28 October 1996 (28.10.96) US 60/052,717 16 July 1997 (16.07.97) US		
(71)(72) Applicant and Inventor: VAN LENDERICH, Bernhard, H. [DE/US]; 18005 33rd Place North, Plymouth, MN 55447 (US).		
(74) Agent: HOLLANDER, Barry, I.; Fisher, Christen & Sabol, Suite 300, 1019 19th Street, N.W., Washington, DC 20036 (US).		Published With international search report.

(54) Title: EMBEDDING AND ENCAPSULATION OF CONTROLLED RELEASE PARTICLES

(57) Abstract

Controlled release, discrete, solid particles which contain an encapsulated and/or embedded component such as a heat sensitive or readily oxidizable pharmaceutically, biologically, or nutritionally active component are continuously produced without substantial destruction of the matrix material or encapsulant. A release-rate controlling component is incorporated into the matrix to control the rate of release of the encapsulant from the particles. The additional component may be a hydrophobic component or a high water binding capacity component for extending the release time. The plasticizable matrix material, such as starch, is admixed with at least one plasticizer, such as water, and at least one release-rate controlling component under low shear mixing conditions to plasticize the plasticizable material without substantially destroying the at least one plasticizable material and to obtain a substantially homogeneous plasticized mass. The plasticizer content is substantially reduced and the temperature of the plasticized mass is substantially reduced prior to admixing the plasticized mass with the encapsulant to avoid substantial destruction of the encapsulant and to obtain a formable, extrudable mixture. The mixture is extruded through a die without substantial or essentially no expansion and cut into discrete, relatively dense particles. Release properties may also be controlled by precoating the encapsulant and/or coating the extruded particles with a film-forming component.

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DETAILED DESCRIPTION OF THE INVENTION

An active component is encapsulated and/or embedded in a plasticizable matrix component or material in an continuous process to produce discrete, solid particles. Release of the active component from the matrix is delayed or controlled over time so that the active component is delivered when and where it is needed to perform its intended function. The release of the active component from the matrix may be controlled by an additional ingredient or additive which affects the hydrophobicity of the matrix or particle, the water binding capacity of the matrix or particle, the solubility or porosity of the matrix material or particle, or the glass transition (T_g) of the matrix material or particle. Pretreatment of the encapsulant, process conditions, and the final shape of the discrete particles may also be used to control the release of the active component from the matrix material. The continuous process of the present invention advantageously at least substantially uniformly distributes, embeds, or encapsulates the active component in the matrix material. The active components may be dispersed in the matrix material on a microscopic or molecular level. Active components which are dispersed on a molecular level may provide a higher bioavailability when released, as compared to their crystalline forms. The active components may be encapsulated or embedded either in a solid form or liquid form. The encapsulants and encapsulated products of the present invention may be edible such as pharmaceutically or biologically or nutritionally active components, or flavors or fragrances or they may be inedible compositions such as a detergent, herbicide, fungicide, pesticide, insecticide, or rodenticide, and the like. Release of the encapsulant from the matrix material may also be controlled by the use of a film or coating upon the encapsulant and/or upon the discrete, solid matrix-encapsulated particles.

The matrix material may be a plasticizable biopolymer such as a carbohydrate, such as a starch or cyclodextrin, or polymer such as polyvinylpyrrolidone or other non-hydrophobic polymers such as copolymers of N-vinylpyrrolidone (NVP) and vinylacetate, polyvinyl alcohol, cellulose esters, cellulose ethers, and polyethylene glycol. Exemplary starches which may be used

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in the present invention are native or modified 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.

5 Additional matrix components which may be used include carbohydrates which have a lower molecular weight than starches. The lower molecular weight matrix components tend to dissolve more readily than does the 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
10 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 dextrans or syrups with dextrose equivalent values (DE values) ranging from about 2 to about 99, or from about 5 to 98, and mixtures thereof.

15 The matrix material is used in an effective encapsulating amount. In embodiments of the present invention, the matrix material content, such as the starch content of the particles may be at least about 40% by weight, for example from about 60% by weight to about 95% by weight, based upon the weight of the final product.

20 The plasticizer or softener which may be used to lower the melt temperature or glass transition temperature (T_g) of the matrix material and facilitate plastification is preferably water but may be an aqueous-based composition such as a sugar solution, alcohol, sorbitol, polyethylene glycol, polypropylene glycol, silicone, hexanol, pentanol, dimethylsulfoxide (DMSO),
25 hexane, or an oil. The amount of plasticizer, such as water, should be sufficient to substantially reduce the melt or glass transition temperature of the plasticizable material such as starch so that it may be admixed with the other ingredients at a sufficiently low temperature and under sufficiently low shear conditions so as to avoid substantial mechanical or thermal destruction of the plasticizable material or
30 matrix material. Exemplary amounts of plasticizer, such as water, may range from about 20% by weight to about 50% by weight, preferably from about 35% by

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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 1 mm to about 3 mm.

5 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.

10 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
15 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
20 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
25 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
30 shelf life. For example, the pellets may be dried to achieve a storage stability or shelf life of at least about nine months, preferably at least about eighteen months,

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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 90°C, preferably from about 20°C to about 60°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, for example, from about 6% by weight to about 9% by weight. In embodiments where no starch or substantially no starch is used as a matrix material, the moisture content may be less than about 6% by weight.

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 or 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 through a sheeting die into a sheet. The extruded sheet may then be cut or molded into individual pieces, such as 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 comprise discrete particles which may be spherical, lens-shaped, or flat discs having diameters of from about 0.5

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What is claimed is

1. A method for continuously encapsulating or embedding a component in a matrix comprising:

- 5 a. admixing at least one plasticizable matrix material with at least one plasticizer and at least one component for controlling the rate of release of an encapsulant, said admixing being under low shear conditions to plasticize the plasticizable material without substantially destroying the at least one plasticizable material and to obtain a substantially
- 10 homogeneous plasticized mass,
- b. reducing the plasticizer content of said plasticized mass,
- c. reducing the temperature of the plasticized mass to obtain a cooled, plasticized mass,
- 15 d. admixing at least one encapsulant with said cooled, plasticized mass to obtain a formable mixture, said admixing being under low shear mixing conditions at a plasticized mass temperature which avoids substantial thermal destruction of said at least one encapsulant,
- 20 e. extruding said formable mixture through a die to obtain an extrudate, and
- f. cutting said extrudate into pieces.

25 2. A method as claimed in claim 1 wherein said plasticizable material and said plasticizer are admixed and heated above the melt or glass transition temperature of the plasticizable material and then admixed with said at least one release-rate controlling component.

3. A method as claimed in claim 1 wherein said plasticizable material, said plasticizer, and said at least one release-rate controlling component are admixed and then heated above the melt or glass transition temperature of the plasticizable material.

United States Patent [19]

Markussen et al.

[11] 4,106,991

[45] Aug. 15, 1978

[54] ENZYME GRANULATE COMPOSITION
AND PROCESS FOR FORMING ENZYME
GRANULATES

[75] Inventors: Erik Kjaer Markussen, Vaerloese;
Arne Wintherhalter Schmidt,
Skovlunde, both of Denmark

[73] Assignee: Novo Industri A/S, Bagsvaerd,
Denmark

[21] Appl. No.: 810,884

[22] Filed: Jun. 28, 1977

[30] Foreign Application Priority Data

Jul. 7, 1976 [GB] United Kingdom 28343/76

[51] Int. Cl.² C07G 7/02

[52] U.S. Cl. 195/63; 195/68

[58] Field of Search 195/63, 68, DIG. 11;
252/DIG. 12

[56] References Cited

U.S. PATENT DOCUMENTS

3,723,327 3/1973 Van Kampen et al. 196/63 X
3,775,331 11/1973 Borrello 195/68 X
4,016,041 4/1977 Van Kampen 195/68

Primary Examiner—Lionel M. Shapiro
Attorney, Agent, or Firm—Fidelman, Wolffe, and
Waldron

[57] ABSTRACT

Improved formation of enzyme granulates through
inclusion within the composition of finely divided cellu-
lose fibres.

Optionally a waxy substance can be employed for the
granulating agent, or to coat the granulate.

11 Claims, 1 Drawing Figure

A preferred embodiment of the process according to the invention comprises a process wherein water is the only granulating agent. By use of this embodiment a relatively cheap granulate with a satisfactory low dust level is produced.

A preferred embodiment of the process according to the invention comprises the use of water and waxy substance as the granulating agents. By use of this embodiment the following advantages are obtained. Due to the fact that water is used as a constituent of the granulating agent the product is relatively cheap. Due to the fact that a waxy substance constitutes a constituent of the granulating agent, the single granules will attain a plastic nature to the point that upon local compression they will not crush and thereby create dust, but will be transformed each into a small flat disc which practically does not give off any dust.

A preferred embodiment of the process according to the invention comprises a granulation carried out at 50-70° C. By use of this embodiment granules with a more homogeneous particle size distribution are produced. In choosing granulation temperature, due regard has to be taken of the heat stability of the enzyme being granulated, some enzymes having a better heat stability than others.

A preferred embodiment of the process according to the invention comprises subjecting the finished granules to coating with a melted wax, preferably polyethylene glycol (PEG), whereafter the thus coated particles optionally are powdered with a finely comminuted coloring agent, preferably TiO₂. This coating can be carried out in any conventional manner, e.g. as described in British Pat. No. 1,362,365, page 1, line 82 to page 2, line 34, and British Patent application No. 34973/73 and 10842/74 corresponding to Belgium Pat. No. 146,802.

Also, the invention comprises the enzyme granulate produced by means of the process according to the invention. Desirably the dry granules have a diameter between 0.3-1.5 mm.

In case the enzyme is used as an enzyme additive for detergents a whitening agent, for example TiO₂, can be incorporated in the granules. By adding the TiO₂ at different times during the granulating process, if the granulating is performed discontinuously, or at different positions in the granulator, if the granulating is performed continuously, the TiO₂ may be distributed inside the granules, or on the surface of the granules or both, as desired.

Preferably all the solid materials are added first to the granulator, whereafter a homogeneous mixture is created and then the granulating agent is introduced as a spray (from one or more of the nozzles present on the granulator).

Usually the filling volume of the total solid starting materials is below 50% of the total volume of the granulator, preferably below 30% of the total volume of the granulator, which, of course, leaves space for the granulating agent.

Surprisingly it has been found that the size of the granules increases much less with time with the fibrous cellulose in the granules than without the fibrous cellulose in the granules. Thus, the granulation can be controlled much easier with the fibrous cellulose than without. The drawing, to which reference is now made illustrates granule growth rates in a granulator according to practice of this invention, as compared to prior art practices.

With the granulation according to practice of the invention it is possible to avoid excessive recirculation of granules which are too fine and too large; actually only about 20% of the granules are recirculated as an average.

Practice of the invention is illustrated in the following specific examples. All the examples have the following in common.

1. The composition of a given composition as a dry powder.

2. Mixing of the dry powder composition.

3. Wetting of the powder mixture with granulating agent e.g. water or a water/binder solution.

4. Processing of the wet powder mixture with the granulating apparatus (rotating knife) until the granulate has the desired particle distribution and degree of roundness.

In all the experiments described in the examples a cylindrical Lodge type mixer FM 130 D I Z (U.S. Pat. No. 3,027,102) has been used. The mixer is equipped with both plough shaped mixers mounted on a horizontal (axial) rotating shaft and a granulating device, consisting of one or more cross knives mounted on a shaft introduced into the mixer through the cylindrical wall in a direction perpendicular to the above mentioned horizontal rotating shaft (i.e. radial of the cylinder).

5. Fluid bed drying of the moist granulate until a dryness which satisfies both the requirements of enzyme stability and the requirements of free-flowing properties and mechanical strength. Usually this will correspond to a water content less than 10%, preferably less than 3%.

In the instances where the granulating agent is exclusively or principally a waxy substance only cooling may be required.

6. Optionally coating.

EXAMPLE I

25% ALCALASE, 10% cellulose fibres, 1% binder: PVP K 30

1. Powder components; 7.5 kg ground proteolytic enzyme ALCALASE (7.5 AU/g) 0.6 kg titanium dioxide 3.0 kg cellulose powder-CEPO S 20 (The Swedish cellulose powder and Wood Flour Mills Ltd.) 18.6 kg ground sodium chloride.

2. The above components are mixed on the Lodge mixer FM 130 D I Z with a rotating speed of the mixer of 160 rpm and with a revolution speed of a single cross knife granulating device of 3000 rpm for 1 minute.

3. Thereafter wetting is performed with 6.6 kg of a 4.5% aqueous solution of polyvinylpyrrolidone (PVP K 30) during continuous mixing with both mixing-aggregate and granulating device.

A pneumatic atomizing nozzle is used, which is adjusted to a 10 minute spraying time.

4. After spraying of the binder solution (according to 3), the moist mixture is further exposed to the compacting action of the granulating device for 8 minutes.

The rotating speed on the mixing aggregate is kept on 160 rpm and on the granulating device on 3000 rpm.

After the treatment a uniform globular to a lens-formed granulate is obtained.

The mixer shows no build-up of an unwanted layer at the end of the process.

5. The moist granulate is dried on a fluidized bed until a moisture content below 3% is obtained.

6. The particle size distribution for the dried granulate is:

6.5%	> 1.4 mm	$d_m = 600\mu\text{m}$
11.5%	> 1.2 mm	
27%	> 840 μm	
39%	> 707 μm	
49%	> 593 μm	
60%	> 500 μm	
75%	> 420 μm	$(d_m \text{ is a symbol designating the average diameter by weight and an abbreviation of diameter mean})$
5.9%	< 300 μm	

EXAMPLE II

(comparative example without fibrous cellulose powder)

25% Alcalase, 1% binder: PVP K 30).

1. Powder components: 7.5 kg ground ALCALASE (7.5 AU/g) 0.6 kg titanium dioxide 21.6 kg ground sodium chloride

2-3. The above composition is mixed and wetted with 3.5 kg of a 8.6% solution of PVP K 30, corresponding to 1% in the final composition, as described in Example 1.

The moist mixture is further exposed to the action of the granulating device for 5 minutes under conditions as described in Example 1.

At the end of the processing a build-up of a hard layer on the wall and tools of the mixer was observed.

5. The moist granulate was dried as described in Example 1.

6. The particle size distribution of the dried granulate is:

6.0%	> 1.4 mm	$d_m = 580\mu\text{m}$
21%	> 840 μm	
30%	> 707 μm	
67%	> 500 μm	
85%	> 420 μm	
3.0%	< 300 μm	

The consequence of incorporating cellulose fibres in connection with the mechanical stability of the granulate was tested by comparing the degradation and formation of fines/dust when the granulate from Example 1 and 2 were treated in a ball mill.

PROCEDURE FOR BREAK-DOWN OF THE GRANULATE

60 g sieved granulate with a particle distribution of 300-840 μm is rotated in a ball mill, which is a closed steel cylinder (diameter 11.5 cm, height 10 cm) with a speed of 100 rpm. The cylinder contains eight steel balls with a diameter of 1.9 cm.

Samples from Example 1 and 2 were treated in this way for 5, 10, 20 and 40 minutes.

After this treatment the mechanical resistance of the granulate is tested according to two procedures.

Procedure 1

The 60 g of the material, which has been exposed to the ball mill treatment, is transferred quantitatively to an elutriation tube, length 2 meter, diameter 35 mm. In the bottom of this tube a sintered glass plate is mounted, on which the sample is placed, whereafter fluidizing with air at a speed of 0.8 m/sec is performed for 40 minutes.

The dust which is blown off and which has a particle size less than about 150 μm , dependent on the roundness of each single particle, is collected quantitatively on a

glassfibre filter, whereafter the dust is weighted and analysed for enzymatic activity.

Procedure 2

The material which has been exposed to the ball mill treatment is transferred quantitatively to a set of sieves, in the actual case 600 μm , 420 μm , 300 μm and 150 μm sieves were chosen whereafter the changes in the particle distribution, caused by the mechanical treatment, are determined.

The granulate according to Examples 1 and 2, compared by procedure 1.

Duration of treatment in ball mill	Experiment 1 (with cellulose fibres)	Experiment 2 (without cellulose fibres)
0 minutes (untreated)	total dust 14.1 mg active dust 3077 μg at 1.5 AU/g	16.8 mg 4143 μg at 1.5 AU/g
5 minutes	total dust 47.4 mg active dust 26,060 μg at 1.5 AU/g	696 mg 662,000 μg at 1.5 AU/g
20 minutes	total dust 1.4 g active dust 1,290,000 μg at 1.5 AU/g	3.9 g 6,100,000 μg at 1.5 AU/g

It appears from the above comparison that the granulate with cellulose fibres releases less dust, both with respect to the total amount and with respect to enzymatic activity, than the preparation without cellulose and leads to a conclusion that cellulose stabilizes the granulate structure.

The granulate according to Examples 1 and 2, compared by procedure 2.

Example 1 With Cellulose Fibres					
Ball Mill Time, min	0	5	10	20	40
% < 840 μm	100	100	100	100	100
% < 600 μm	66.0	65.8	69.5	72.4	72.3
% < 420 μm	25.3	28.4	32.6	36.4	40.1
% < 300 μm	~0	2.6	5.0	8.6	17.8
% < 150 μm	~0	0.03	0.15	2.3	11.0

Example 2 Without Cellulose Fibres					
Ball Mill Time, min	0	5	10	20	40
% < 840 μm	100	100	100	100	100
% < 600 μm	64.0	70.4	88.9	96.3	99.85
% < 420 μm	15.8	34.7	57.4	72.3	93.1
% < 300 μm	~0	8.6	23.9	39.7	64.6
% < 150 μm	~0	0.47	2.4	6.8	17.2

It appears from the tables that the granulate without cellulose fibres is broken down more quickly and releases more dust (< 150 μm) during ball mill treatment.

EXAMPLE III

(Composite as Example 1, with change of apparatus parameters)

A granulate is prepared as in Example 1, with the difference, that the mixing device is adjusted to 120 rpm during the granulation and instead of a pneumatic nozzle a pressure nozzle is used.

A four cross knives granulating tool was employed. Particle size distribution for the dried granulate was as follows:

1.3%	> 1.4 mm
8.3%	> 1.0 mm
16%	> 840 μ m
37%	> 710 μ m
49%	> 600 μ m
71%	> 500 μ m
84%	> 420 μ m
6%	< 300 μ m

 $d_m = 600 \mu\text{m}$ **EXAMPLE IV**

(25% ALCALASE, 10% cellulose fibres 10% binder; yellow dextrine)

1. Power Components:

7.5	kg	ground ALCALASE - 7.5 AU/g
15.9	kg	ground sodium chloride
3.0	kg	yellow dextrine
0.6	kg	titanium dioxide
3.0	kg	fibrous cellulose powder (CEPO S 40)

2. The above composition is mixed as described in Example 1, employing in this instance 3.0 kg of water sprayed on the mixture.

3. The mixture is granulated for 4 minutes, as described in Example 1.

4. The granulate is dried as described in Example 1.

5. Particle size distribution for the dried granulate:

10%	> 1.2 mm
24%	> 840 μ m
34%	> 707 μ m
44%	> 595 μ m
79%	> 420 μ m
12%	< 300 μ m

 $d_m = 590 \mu\text{m}$ **EXAMPLE V**

(25% ALCALASE, 15% cellulose fibres, 2% binder: hydroxypropylcellulose).

1. A composition consisting of 4 kg ground ALCALASE - 7.5 AU/g 12.2 kg ground sodium chloride 0.4 kg titanium dioxide 3.0 kg fibrous cellulose CEPO S 40 (15%)

2. is mixed according to example 1, whereafter

3. 6.4 kg of a 7% solution of hydroxypropylcellulose KLUCCEL E is sprayed on the mixture according to example 1. (The word KLUCCEL is a Trademark).

4. The moist mixture is granulated for 9 minutes, and otherwise example 1 is followed.

5. The granulate is dried according to Example 1.

6. Particle size distribution for the dried granulate:

16.0%	> 840 μ m
29%	> 707 μ m
62%	> 500 μ m
78%	> 420 μ m
5.3%	< 300 μ m

 $d_m = 570 \mu\text{m}$ **EXAMPLE VI**

A composition according to Example 1 is prepared and wetted with 7.0 kg of a 4.3% solution of PVP K 30.

The wetted mixture is further granulated for 6 minutes. During the granulation samples are taken after 2, 3 and 4 minutes. Drying is performed according to example 1.

The particle size distribution for the dried granulate after the granulation treatment in 2, 3, 4 and 6 minutes, respectively, is as follows:

	2 min	3 min	4 min	6 min
> 1.2 mm	5.5	6.0	9.2	10.2
> 840 μ m	18	20	25	30
> 707 μ m	29	31	38	45
> 500 μ m	61	66	78	80
> 420 μ m	81	86	89	93
> 300 μ m	2.2	1.4	1.5	0.9
d_m	550 μ m	580 μ m	625 μ m	670 μ m
$d_m(t)/d_m(4)$	0.88	0.92	1	1.07

$d_m(t)$ is the average diameter after t minutes of granulation. $d_m(t)/d_m(4)$ is a growth parameter chosen to illustrate growth versus time.

EXAMPLE VII

(Composition as Example 1). Examples 6 and 7 show the growth of the particle as a function of the duration of the granulation.

A composition according to Example 1 is prepared and wetted with 6.0 kg of a 5% solution of PVP K 30 action.

The wetted mixture is further granulated for 12 minutes; during the granulation samples are taken after 4, 6 and 8 mins.

The particle size distribution for the dried granulate after the granulation treatment in 4, 6, 8 and 12 minutes, respectively, is as follows:

	4 min	6 min	8 min	12 min
> 1.2 mm	3.9	4.1	3.9	5.0
> 840 μ m	12	14	15	17
> 707 μ m	19	22	23	27
> 500 μ m	39	46	53	56
> 420 μ m	53	63	64	77
> 300 μ m	17	8.9	10.0	6.7
d_m	435	475	485	515
$d_m(t)/d_m(4)$	1	1.09	1.12	1.18

EXAMPLE VIII

(Comparative example without fibrous cellulose powder). Example 8 and 9 are comparative examples to Examples 6 and 7.

A composition is prepared and wetted as described in example 2 with 3.9 kg of a 7.7% PVP K 30 solution.

The wetted mixture is further granulated in 2, 4 and 6 minutes respectively, and is dried according to example 1.

The particle size distribution of the dried granulate after the granulating treatment of 2, 4, and 6 minutes, respectively, is as follows:

	2 min	4 min	6 min
> 1.4 mm	3.8	11	11
> 1.0 mm	10	25	39
> 840 μ m	16	41	64
> 707 μ m	24	58	82
> 500 μ m	51	88	96
> 420 μ m	74	96	98
> 300 μ m	5.2	1.4	1.1
d_m	510	770	920 μ m
$d_m(t)/d_m(4)$	0.66	1	1.19

EXAMPLE IX

(Comparatively example without fibrous cellulose powder).

A composition is prepared and wetted as described in Example 2 with 3.5 kg of a 8.6% aqueous PVP solution.

5. The granulate is cooled in a mixer with gentle agitation whereby it solidifies to a somewhat sticky granulate, with the stickiness ascribed to the CMEA.

6. Particle size for the cooled granulate is as follows:

> 1.7 mm	9.0%
> 1.4 —	17%
> 1.2 —	24%
> 1.0 —	41%
> 840 μ m	65%
> 600 —	93%
< 420 —	0.5%

$d_m = 940 \mu$ m

EXAMPLE XX

(25% Alcalase, 10% cellulose fibers, 18% CMEA)

1. Powder composition: 7.5 kg ground Alcalase @ concentrate (7.4 Anson units/g) 0.6 kg titanium dioxide 3.0 kg fibrous cellulose (ARBOCEL @ BSM 300) 13.5 kg ground sodium chloride

2. The above components are mixed and heated to 70° C.

3. The mixture is sprayed with 5.4 kg melted CMEA as described in Example 19, the spraying time being adjusted to 4 minutes. Thereafter the spraying is continued with 2.6 kg water, the spraying time being adjusted to 2 minutes. The mixing device is operated at 95 rpm during the spraying and the granulating device at 3000 rpm.

4. After spraying the mixture is further compacted for 6 minutes with the mixing device at 175 rpm and the granulating device at 3000 rpm.

5. The granulate is dried as described in Example 1, whereafter it is cooled to 30° C. Now the granulate appears as a free flowing granulate.

6. The particle size distribution is as follows:

0.2%	> 1.7 mm
2.2%	> 1.4 mm
15%	> 1.0 mm
35%	> 841 μ m
72%	> 600 μ m
92%	> 420 μ m
3.9%	< 3.9 μ m

$d_m = 730 \mu$ m

EXAMPLE XXI

(25% Alcalase, 20% cellulose fibers, 20% PEG 1500)

1. Powder composition: 5 kg ALCALASE @ (7.4 Anson units/g), ground 0.4 kg titanium dioxide 4.0 kg fibrous cellulose CEPO S 20 6.6 kg ground sodium chloride

2. The above components are mixed and heated to 55° C as described in Example 18.

3. The mixture is sprayed with a solution consisting of 4 kg polyethylene glycol 1500 and 2.5 kg of water, the solution being kept at 55° C and the spraying time being adjusted to 7 minutes. The mixing device is operated at 95 rpm during the spraying and the granulating device at 3000 rpm.

4. After spraying the mixture is further compacted for 8 minutes with the mixing device at 175 rpm and the granulating device at 3000 rpm.

5. The granulate is dried as described in Example 1 whereafter it is cooled to 30° C. Now the granulate appears as a free flowing granulate.

6. The granulate has the following particle size distribution:

2.1%	> 1.2 mm
8.4%	> 1.0 —
20%	> 841 μ
52%	> 600 μ
29%	> 420 μ
6.6%	< 300 μ

$d_m = 610 \mu$

EXAMPLE XXII

1,2,3,4,5: as in Example 1.

5a 7 kg granulate as prepared in Example 1 and after a sieving procedure where particles greater than 840 μ and smaller than 300 μ m have been removed, is heated to 55° C in a jacketed Lodge mixer M 20.

The hot granulate is sprayed with 7% polyethylene glycol 1500 (60° C) with continuous mixing. After distribution of PEG 1500 the granulate is powdered with 8.5% titanium dioxide with continuous mixing, TiO₂ being used as a whitening agent.

After distribution of TiO₂ a further 2% PEG 1500 is supplied in order to stick all the powder to the surface of the granulate.

All percentages are based on the weight of the dry uncoated granulate.

Half of the hot coated granulate is cooled in the mixer using gentle agitation and cooling water in the jacket.

The other half of the hot coated granulate is transferred to a cooler with rotating cooling coils.

After cooling the granulate is further sieved between 300 and 840 μ m.

EXAMPLE XXIII

1,2,3,4,5: As in Example 1.

5a. 7 kg granulate as prepared in Example 1 is heated to 70° C in a jacketed Lodge M 20.

The hot granulate is sprayed with 13% PEG 6000 (in which 0.2% of a blue dye polar brilliant blue RAWL, Ciba Geigy is dispersed) during continuous mixing. All percentages are based on the weight of the dry, uncoated granulate.

After homogeneous distribution of the color the granulate is cooled and sieved as described in Example 22.

EXAMPLE XXIV

Example 23 was repeated except that the dye was powdered directly on the base granulate, whereafter the coating with PEG was performed.

What is claimed:

1. In the process for drum granulating an enzyme composition including enzyme, inorganic salts, and a granulation binder, with a liquid phase granulating agent, the improvement which comprises incorporating into the composition undergoing granulation finely divided cellulose fibers in an amount of 2-40% w/w based upon the dry weight of the total composition.

2. The process of claim 1 wherein the cellulose has an average fiber length of 50-160 μ and an average fiber width of 20-30 μ .

3. The process of claim 1 wherein the fibrous cellulose is 5-30% w/w.

4. The process of claim 1 wherein the granulating agent is water.

5. The process of claim 1 wherein the granulating agent is a mixture of water and a waxy substance.

6. The process of claim 1 wherein granulation is performed at 50°-70° C.

United States Patent [19]

Bogerman et al.

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4,242,219

[45]

Dec. 30, 1980

[54] NOVEL ENZYME PARTICLES AND THEIR PREPARATION

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435/188

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252/174.12; 195/62, 63, 66 R, 68; 435/177, 178,
187, 188; 424/94, 31, 32, 33, 35, 38

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[57]

ABSTRACT

A novel process for preparing non-dusting, enzyme-containing particles, e.g. for introduction into washing compositions, comprising homogeneously mixing an amount of an enzyme in dry or substantially dry form to give the desired enzyme activity in the particles when formed, about 20% to about 60% of a hydrophilic organic cohesive compatible with the enzyme, about 10% to about 60% of a building agent suitable for use in enzyme compositions, and a sufficient amount of water, if necessary, to give a moisture content in the particles, when formed, of from 5% to 15%, mechanically dividing the mixture obtained into particles of the desired size, and coating the particles to prevent loss of moisture and the novel enzyme particles produced thereby as well as washing compositions and pharmaceutical compositions containing said enzyme particles.

33 Claims, No Drawings

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NOVEL ENZYME PARTICLES AND THEIR PREPARATION

STATE OF THE ART

Enzymes have been used in washing compositions for several years but one of the problems in the formulation of enzymatically active washing compositions is the fact that, when formulating the enzymatic washing compositions enzyme dust is formed. This is disagreeable to the people working with the enzyme compositions and sometimes, skin irritations or allergic reactions occur.

Dusting of the enzyme may be avoided to a considerable extent by changing the enzyme into a form other than a powder in which it is normally obtained and this is advantageously done by converting the enzyme into a solid particulate form. Processes for changing enzymes into a particulate form are known, for example, as described in British Pat. No. 1,324,116 which discloses a process wherein a liquid mixture of the enzyme and a binder such as a low-melting nonionic emulsifier is dispersed into droplets by a toothed disc, and is guided into a cooling tower wherein the droplets are allowed to solidify. In this manner, a dust-free product is obtained, which is easily formulated into washing compositions.

Although the enzyme particles obtained according to the said British patent are good and may be used without risk of dust formation, they have shown the disadvantage that, if mechanical pressure is exerted upon the particles, e.g. the foot pressure of somebody walking over spilled particles, they easily break down into smaller particles and powder so that enzyme powder from the broken particles may be carried along with air currents.

OBJECTS OF THE INVENTION

It is an object of the present invention to provide pliant enzyme particles that do not, or substantially do not, break down to powder when mechanical pressure is applied to them.

It is a further object of the invention to provide washing and pharmaceutical compositions containing dust-free, enzyme containing particles.

It is another object of the invention to provide a novel process for the preparation of dust-free, enzyme containing particles which are resistant to mechanical breakage.

These and other objects and advantages of the invention will become obvious from the following detailed description.

THE INVENTION

The novel process of the invention for the preparation of enzyme-containing particles which do not, or substantially do not, dust when external mechanical pressure is applied to them comprises homogeneously mixing an amount (preferably up to about 50%) of an enzyme in dry or substantially dry form sufficient to give the desired enzyme activity in the particles when formed, about 20 to about 60%, (preferably about 30% to about 50%) of a hydrophilic organic cohesive compatible with the enzyme, about 10% to about 60% (preferably about 20% to about 45%) of a building agent suitable for use in enzyme compositions, and a small amount of water, if necessary, sufficient to give a moisture content in the final particles when formed of from 5% to 15%, mechanically dividing the mixture obtained

into particles such as pellets, of desired size, and coating the particles so as to prevent loss of moisture.

It may not be necessary to add water if the moisture content of the components is sufficient to supply the required moisture content in the final product. There may optionally be added an amount of a moisture-regulating agent sufficient to maintain a moisture content in the particles when formed of from about 5% to about 15% at ordinary temperatures and ordinary relative humidities.

The percentage mentioned in this specification and the accompanying claims are percentages by weight, unless otherwise indicated.

For the washing compositions of the invention, the basic formulation may be selected from a wide range of raw materials since they need not be consumable, although they preferably are biodegradable. For pharmaceutical compositions destined to be administered orally, the components should be edible and pharmaceutically acceptable.

The enzyme-containing particles obtained by the invention are extremely useful for washing compositions if, for the purpose, the enzymes are selected from those customarily used in washing compositions. Examples of these enzymes are proteolytic enzymes (proteases), amylolytic enzymes (amylases), or lipolytic enzymes (lipases) and mixtures thereof. Examples of suitable proteolytic enzymes are enzymes from bacterial origin, such as proteolytic enzymes from *Bacillus* species, e.g. *B. subtilis*, *B. licheniformis* and *B. alcalophilus*. Amylolytic enzymes are preferably also enzymes of bacterial origin, such as amylolytic enzymes produced by *Bacillus* species, e.g. *B. subtilis*. Some bacteria produce suitable proteolytic and amylolytic enzymes simultaneously. Lipolytic enzymes which may be used are preferably also enzymes of microbial origin. They may be produced by *Candida* species, e.g. *Candida cylindracea*. Lactases are also useful. Enzymes useful for pharmaceutical compositions are, e.g. invertase, glucosylase etc.

The hydrophilic organic cohesive used in the formation of the enzyme mix may be selected from starch and cellulose derivatives, and a preferred type of starch derivative is acetylated amylopectin called "amylopectin gum". This type of gum is an amylopectin with an acetylation degree of about 5% to about 30%. Examples of other useful cohesives are gum arabic, dextrin, karaya gum, methylcellulose, tragacanth and other types of amylopectin, such as methylamylopectin. Certain starch hydrolysates preferably having a DE (Dextrose Equivalent) of about 30 may also be used. A starch hydrolysate may be used as cohesive if it has cohesive properties, i.e. if it contains amylopectin derivatives.

The building agent included in the enzyme mix is preferably a water-soluble carbohydrate, generally containing from 5 to 12 (preferably 6) carbon atoms. Suitable examples of carbohydrates having 5 carbon atoms—the pentoses—are xylose, arabinose, ribose and lyxose. Suitable examples of carbohydrates having 6 carbon atoms (the hexoses) are glucose, mannose, galactose, fructose and sorbose. Furthermore, disaccharides, normally having 12 carbon atoms, may be used, such as sucrose, maltose, or cellobiose. The most preferred carbohydrate is glucose. As glucose crystallizes rather easily as do several other carbohydrates, a part, or all of the glucose or other carbohydrate may be replaced by starch hydrolyzed to a certain extent. A starch hydroly-

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-continued

Ingredients	Sample Number						
	1	2	3	4	5	6	7
Elutriation test (3)	29	20	14	10	20	32	11
(1) 2.18 million DU/g (2) starch hydrolysate, about 43 DE (3) for 60 grams of sample.							

The said granules had good properties.

EXAMPLE 11

The following recipe containing an alkaline protease was prepared: 34% Amylogum CLS-0473, 17% of glucose, 17% of glucose syrup (starch hydrolysate, about 43 DE), 7% of stearic acid, 20% of PB 92 (1.7 million ADU/g) (1) and 5% of titanium dioxide.

A granulate was made by the method of Example 5 with coating of the granulate carried out as in Example 3. The granulate proved to have similar properties: inter alia the elutriation test applied on a sample of 60 g showed a value of 95 ADU/g. (1) PB 92 is an alkaline proteolytic enzyme described in U.S. Pat. No. 4,002,572.

EXAMPLE 12

Storage stability of the granulate

400 g of a commercially available washing powder containing sodium perborate were added to an amount of enzyme granulate of Example 10 (Sample 5) previously coated with 1% of a 1:1 mixture of glycerol monostearate and paraffin oil, to make the activity 6,000 DU/g and the composition was mixed thoroughly again. Weighed samples of about 20 g taken from the mixture were analyzed for a storage stability test just after mixing and after 0.5, 1, 2 and 3 months storage at 37° C. The samples were tested by dissolving them in synthetic tap water (containing 111 mg/l of CaO, 27.5 mg/l of MgO and 210 mg/l of NaHCO₃, German Hardness 15°), containing sufficient sodium dithionite to neutralize the action of the perborate, and diluting with synthetic tap water to a concentration suitable for testing the activity in Delft Units (cf. British Pat. No. 1,353,317) (series A in the following Table).

A similar test was run with a commercially available granulate of the same enzyme (Maxatase P, prepared by the method described in British Pat. No. 1,324,116) (series B.). The following results were obtained:

Storage time (in months)	0.5	1	2	3
Series A	100	99	89	91
Series B	94	92	82	75

The Table shows that the storage stability of series A of the invention is good and is even better than that of the comparative series B, which also had a fairly good stability.

EXAMPLE 13

A preparation containing lactase was prepared as follows: 45% of Amylogum CLS-0473, 30% of lactase (precipitated) and 25% of glucose syrup (starch hydrolysate, about 43 DE). A granulate was made by the method of Example 5 and was coated as described in Example 3. When the temperature was kept at 50° C. or below during preparation of the granules, the activity of the granulate was 95% of the starting activity, whereas, when the temperature was allowed to rise to 50° to 70°

C. the remaining activity was considerably lower, i.e. 55% of the original activity. This Example shows that the temperature to be used depends greatly on the enzyme.

EXAMPLE 14

The following recipe was used for the preparation of particles by the procedure described in Example 1: 1 part by weight of glycerol, 2 parts by weight of polyvinylpyrrolidone, 2 parts by weight of Berol 80, 1 part by weight of Maxatase, 5 parts by weight of titanium dioxide, 22 parts by weight of glucose syrup (starch hydrolysate of about 43 DE), 21 parts by weight of glucose and 46 parts by weight of Amylogum CLS-0473. The particles were coated as described in Example 1.

Various modifications of the products and process of the invention may be made without departing from the spirit or scope thereof and it should be understood that the invention is intended to be limited only as defined in the appended claims.

What we claim is:

1. A process for the preparation of enzyme-containing particles which do not, or substantially do not, dust when subjected to external mechanical pressure comprising homogeneously mixing an amount of an enzyme in dry or substantially dry form sufficient to give the desired enzyme activity in the particles when formed, about 20% to about 60% of a hydrophilic organic cohesive compatible with the enzyme selected from the group consisting of starch derivatives, cellulose derivatives, arabic gum, karaya gum and tragacanth, about 10% to about 60% of a building agent suitable for use in enzyme compositions selected from the group consisting of water-soluble carbohydrates of 5 to 12 carbon atoms, up to 70% of which may be replaced by a starch hydrolysate of about 20 to about 70 DE, and an amount of a moisture regulating agent selected from the group consisting of polyols having 2 to 6 carbon atoms and 2 to 4 hydroxy groups attached to different carbon atoms sufficient to maintain a moisture content in the particles formed of from 5% to 15% at ordinary temperatures and ordinary relative humidities and sufficient water to give a moisture content in the particles formed of from 5% to 15%, mechanically dividing the mixture obtained into particles of the desired size, and coating the particles to prevent loss of moisture with about 0.5 to about 5% of a water repellent agent selected from the group consisting of paraffin oil, linseed oil, earth wax, cocoa fat, candelilla wax, carnauba wax, paraffin wax and beeswax, ceresine wax and lanolin and mixtures thereof.

2. The process of claim 1 wherein the composition contains up to 50% of the enzyme and 20 to 60% of the building agent.

3. The process of claim 1 wherein the hydrophilic cohesive is selected from the group consisting of acetylated amylopectin, gum arabic, dextrin, karaya gum, methylcellulose, tragacanth, methylamylopectin and starch hydrolysates having cohesive properties.

4. The process of claim 1 wherein the hydrophilic cohesive is acetylated amylopectin with an acetylation degree of about 5 to about 30%.

5. The process of claim 1 wherein the amount of hydrophilic cohesive used is about 30% to about 50%.

6. The process of claim 1 wherein the building agent is selected from the group consisting of water-soluble carbohydrates containing 5 to 12 carbon atoms.

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Lengerich et al.



US005079012A

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[45] Date of Patent: Jan. 7, 1992

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[54] SHELF STABLE COOKIE PRODUCT
CONTAINING HEAT AND SHEAR
SENSITIVE ADDITIVES AND METHOD OF
MAKING

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subsequent to Dec. 31, 2003 has been
disclaimed.

[21] Appl. No.: 489,329

[22] Filed: Mar. 5, 1990

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 362,476, Jun. 7, 1989,
and a continuation-in-part of Ser. No. 362,526, Jun. 7,
1989, and a continuation-in-part of Ser. No. 362,747,
Jun. 7, 1989, and a continuation-in-part of Ser. No.
362,472, Jun. 7, 1989.

[51] Int. Cl.³ A21D 8/00

[52] U.S. Cl. 426/72; 426/74;
426/549; 426/552

[58] Field of Search 426/549, 552, 72, 74

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[57] ABSTRACT

The present invention relates to the production of a shelf stable unleavened cookie composition which can be leavened to exhibit a cookie crumb-like structure after baking in a convection, radio frequency, or microwave oven. The cookie composition is prepared by using a cooker extruder to mix ingredients and to heat the ingredients to preferably at least about 200° F. to promote browning and flavor development. The resulting cookie composition has a water activity of less than 0.7, preferably less than 0.6. The cookie composition produced using the cooker extruder is shaped into cookie preforms, and leavened by heating in an oven such as a microwave, convection oven, or a combination thereof. The total moisture content of the cookie mixture is generally less than 10% and preferably less than 8%. Water or a source of water may be admixed with the cookie mixture in an amount sufficient to form a dough-like consistency. The water may be admixed in the extruder after the heating stage to prevent fat separation or alternatively after the cookie composition has cooled and before use by the consumer. The cookie composition may contain a heat or shear sensitive additive. The additive is admixed with the heat treated cookie composition under conditions such that the additive is not deactivated.

51 Claims, No Drawings

configuration may be used with a model ZPM-120 continuous mixer as the second stage mixing device.

In this embodiment the heat treated mass continuously produced in the extrusion cooker is extruded and transferred to a post extrusion or second stage mixer, where it is cooled and admixed with ingredients to form a shelf stable cookie mixture. The heat and/or shear sensitive additives are admixed with the heat treated mass in the post extrusion mixer to avoid extensive heat treatment and mixing.

The use of substantially the entire maximum available length of the extruder to heat treat the ingredients comprising oil, flour and optionally, at least one sugar permits significant production of Maillard reaction precursors. It further substantially reduces the amount of post extrusion heat treatment required for final baking of the output product. Also, thermal stress on the extruder screws may be reduced by maintaining a more even temperature profile in the extruder.

Generally, in embodiments where a post extrusion mixer is used the heat treated mass is extrusion from the cooker extruder without substantial moisture loss or flashing, such as would cause substantial expansion or puffing, because of the low water content of the mass and the low pressure drop upon exiting the extruder. Suitably, a die is not used with the cooker extruder. The extrudate from the cooker extruder may be dropped or conveyed into the inlet of a continuous post-extrusion mixer.

In embodiments of the present invention where a post extrusion mixer is used, the heat treated mass, upon exiting the cooker extruder, may be further heated to promote further browning and flavor development and to reduce the amount of post extrusion heating needed for final baking of the output product provided that adverse oil separation or extruder surging does not occur. This further heating may be up to an additional 200° F. for a short period of time, e.g. 5-10 seconds. For example, a microwave heating device may be placed between the extrusion cooker and the post extrusion mixer for heating the heat treated mass before it enters the post extrusion mixer. The minimal amount of final baking required due to the use of the entire length of the extruder for heat treatment and the further use of the microwave applicator permits the addition of heat sensitive ingredients to the formulation, for example, through a downstream feed port in the continuous mixer.

A microwave applicator which may be used is disclosed in U.S. patent application Ser. No. 372,384 entitled "Extruder And Continuous Mixer Arrangement For Producing An At Least Partially Baked Product Having A Cookie-Like Crumb Structure The Extruder Including A Microwave Applicator" filed in the name of Bernhard Van Lengerich on June 7, 1989, abandoned in favor of Ser. No. 441,230 filed Nov. 22, 1989. The disclosures of both applications are incorporated herein by reference in their entireties. As disclosed therein, the microwave applicator is formed as a continuation of the screw channel at the downstream most end of the extruder. The screw channel beyond the screw elements and within the microwave applicator is conformed to a round channel surrounded by a source of microwave energy. The residence time of the ingredients within the microwave applicator is relatively short, e.g. about 5-6 seconds.

The present invention is further illustrated in the following examples where all parts, ratios, and percent-

ages are by weight and all temperatures are in °F., unless otherwise stated:

EXAMPLE 1

The ingredients, their feed placement, and their relative amounts used to prepare a shelf stable, extruded, partially baked cookie preform which was leavened into a cookie having a crumb-like structure and texture using dielectric radio frequency heating in accordance with the present invention were:

INGREDIENT AND PLACEMENT	WEIGHT %
<u>Component 1: first dry feed port</u>	
Wheat flour, bleached (about 12% by weight water)	49.88
Non-fat dry milk (about 52% by weight lactose)	1.50
Salt	0.75
<u>Component 2: first dry feed port</u>	
White sugar (sucrose), granulated	14.72
Brown sugar (about 89% sucrose, 3% invert, 4% non-sugar solids, 3% water)	6.86
Vanilla	1.23
Sodium bicarbonate	0.25
<u>Component 3: first liquid feed port</u>	
Soybean spray oil	22.56
<u>Component 4: second liquid feed port</u>	
Tap water	2.25
Total	100.00

The co-rotating twin screw cooker extruder used to prepare the cookie products of the present invention was a Werner and Pfleiderer ZSK 57 equipped with twelve barrels, each provided with external, jacketed heating and cooling means. First and second dry feed ports were set up at barrels 1 and 8, respectively. These two dry feed ports were open to the atmosphere. The first liquid feed port was set up between barrels 1 and 2. The second liquid feed port was the same as the second dry feed port at barrel 8. The second liquid feed inlet pipe was inserted into the open port at barrel 8 so that optional second dry feed ingredients and the second liquid feed ingredients could be separately fed into the same port. Dry ingredients were not fed to the second dry feed port in this example.

The first barrel, which contained the first dry feed port, was set on constant cool. The remaining eleven barrels were divided into 7 separately measured barrel temperature zones. Barrels 2 and 3 corresponded to temperature zone 1, barrel 4 corresponded to zone 2, barrels 5 and 6 corresponded to zone 3, barrels 7 and 8 corresponded to zones 4, and 5, respectively, barrels 9 and 10 corresponded to temperature zone 6, and barrels 11 and 12 corresponded to temperature zone 7. The thermocouples for measuring actual barrel temperatures were located in barrels 2, 4, 5, 7, 8, 10, and 12.

Barrels 2 through 6 were set to heat to 350° F., barrel seven to 200° F., barrel eight was set on constant cool, barrels 9 and 10 were set at 100° F. and barrels 11 and 12 were set at 110° F.

Paddles and screw elements were arranged on the screw shafts from upstream to downstream to provide: a) rapid conveying of the added dry ingredients in barrel 1, b) conveying of the dry ingredients and added oil and gradually increasing the degree of fill in barrel 2, c) chopping or mixing action for admixing the oil and dry ingredients, conveying to gradually increase the degree of fill, and mixing in barrel 3, d) repeated conveying and

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dielectric oven set at about 81% power for 45 seconds to produce distinctly leavened, surface browned cookies having a crumb-like structure and crumb-like texture.

EXAMPLE 3

The ingredients, their feed placement, and their relative amounts which may be used to prepare a shelf stable, extruded, partially baked cookie preform suitable for leavening into a cookie having a crumb-like structure and texture using dielectric radio frequency heating in accordance with the present invention are:

INGREDIENT AND PLACEMENT	WEIGHT %
<u>Component 1: first dry feed port</u>	
Wheat flour, bleached (about 12% by weight water)	45.72
Non-fat dry milk (about 52% by weight lactose)	1.37
Salt	0.69
White sugar (sucrose), granulated	13.72
<u>Component 2: second dry feed port</u>	
Brown sugar (about 89% sucrose, 3% invert, 4% non-sugar solids, 3% water)	6.40
Nut pieces (walnut fluff)	7.31
<u>Component 3: first liquid feed port</u>	
Soybean spray oil	20.58
<u>Component 4: second liquid feed port</u>	
Tap water	4.21
Total	100.00

The co-rotating twin screw cooker extruder system of Example 1 may be used to prepare the cookie products of the present invention.

The screws may be rotated at about 225 rpm at about 5% of maximum torque. The ingredients may be fed in their relative amounts to provide a throughput or mass flow rate of the dough-like mixture extrudate of about 203 lbs/hr.

Component 1 may be prepared by mixing the ingredients to obtain a substantially homogeneous dry blend. The component 1 dry blend may be continuously fed to the first dry feed port. Component 2 may be prepared by mixing the ingredients to obtain a second substantially homogeneous dry blend which may be continuously fed downstream to the second dry feed port. Component 3 may be prepared by melting the semi-solid soybean spray oil to obtain a liquid oil which may be continuously fed to the first liquid feed port. Component 4 may be continuously fed to the second liquid feed port.

On a calculated basis, the water content of the dough-like mixture formed in the extruder is about 10.7% by weight, based upon the total weight of the dough-like mixture.

The barrel set temperatures, pressures, and residence times may be the same as in Example 1.

The substantially homogeneous dough-like mixture formed in the extruder may be extruded through a horizontally oriented slit die (about 5 mm high by about 50 mm wide) to obtain a continuous, substantially unleavened ribbon having a temperature upon exiting the extruder of less than about 150° F. The ribbon may be cut into cylindrical pieces having a diameter of about 1½". The pieces will have a water activity of about 0.7.

Six pieces having an average weight of about 4.2 gm/piece may be subjected to heating in a continuous dielectric oven set at about 81% power for 45 seconds

to produce distinctly leavened, surface browned cookies having a crumb-like structure and crumb-like texture.

EXAMPLE 4

The ingredients, their feed placement, and their relative amounts used to prepare an extruded, partially baked, shelf stable cookie preform which was leavened into a cookie having a crumb-like structure and texture using dielectric radio frequency heating in accordance with the present invention were:

INGREDIENT AND PLACEMENT	WEIGHT %
<u>Component 1: first dry feed port</u>	
Wheat flour, bleached (about 12% by weight water)	49.8
Non-fat dry milk (about 52% by weight lactose)	1.50
Salt	0.75
<u>Component 2: first dry feed port</u>	
White sugar (sucrose), granulated	15.64
Brown sugar (about 89% sucrose, 3% invert, 4% non-sugar solids, 3% water)	7.29
Vitamin mix	0.13
<u>Component 3: first liquid feed port</u>	
Soybean spray oil	22.56
<u>Component 4: second liquid feed port</u>	
Tap water	2.25
Total	100.00

The co-rotating twin screw cooker extruder system of Example 1 was used to prepare the cookie products of the present invention.

The screws were rotated at about 125 rpm at about 3% of maximum torque. The ingredients were fed in their relative amounts to provide a throughput or mass flow rate of the dough-like mixture extrudate of about 203 lbs/hr.

Component 1 was prepared by mixing the ingredients to obtain a substantially homogeneous dry blend. The component 1 dry blend was continuously fed to the first dry feed port. Component 2 was prepared by mixing the ingredients to obtain a second substantially homogeneous dry blend which was continuously fed to the first dry feed port. Component 3 was prepared by melting the semi-solid soybean spray oil to obtain a liquid oil which was continuously fed to the first liquid feed port. Component 4 was continuously fed to the second liquid feed port.

On a calculated basis, the water content of the dough-like mixture formed in the extruder was about 8.5% by weight, based upon the total weight of the dough-like mixture, and the water activity was less than about 0.7.

At steady state, the barrel set temperatures and the actual barrel temperatures were:

Barrel #	Barrel Set Temperature °F.	Actual Barrel Temperature, °F.
1	cool	—
2	350	235
3	350	—
4	350	281
5	350	341
6	350	—
7	200	199
8	<100	92
9	100	—
10	100	104
11	110	—

Water and sugar are fed at a steady rate to the flour and fat slurry and mixed uniformly to form a dough-like consistency. Nut pieces were added to the continuous mixer through the third dry feed port in the barrel at a rate of 125 lbs/hr and uniformly mixed with the dough-like composition. The addition of the water and the sugar contribute to the overall lowering of the temperature of the composition.

Six pieces were subjected to microwaving in a microwave oven for 50 seconds to produce distinctly leavened cookies having dimensions of about 1½" by about 1½". The cookies will be surface browned and have a crumb-like structure and crumb-like texture.

What is claimed is:

1. A method for the continuous production of a shelf stable, unleavened cookie composition comprising:

- (a) admixing ingredients comprising flour, at least one sugar, and shortening or fat in a cooker extruder,
- (b) heating ingredients comprising flour and shortening or fat in the extruder while continuously mixing at a temperature of at least 150° F. to promote browning and to form a heat treated, unleavened cookie composition, and
- (c) extruding the unleavened cookie composition, wherein the unleavened cookie composition includes a pH-adjusting agent and has a pH of about pH 5 to about pH 9.5 and a water activity of less than 0.7, and wherein the extruded cookie composition is leavenable by heating to a crumb-like structure.

2. A method as claimed in claim 1 wherein the water content of said unleavened cookie composition is sufficiently low such that further water addition increases the consistency of the composition.

3. A method as in claim 1 wherein the water content of the unleavened cookie composition is such that the cookie composition is crumbly at room temperature.

4. A method as claimed in claim 1 wherein at least one reducing sugar is admixed with said ingredients comprising flour in an amount to promote Maillard browning during heating.

5. A method as claimed in claim 1 wherein at least one protein source is admixed with said ingredients comprising flour shortening or fat in an effective amount to promote Maillard browning during heating.

6. A method as claimed in claim 1 wherein said unleavened cookie composition comprises:

- (a) less than about 20% by weight water,
- (b) about 12% by weight to about 40% by weight shortening or fat,
- (c) about 10% by weight to about 40% by weight of sugar,
- (d) about 30% by weight to about 70% by weight of at least one flour,

said percentages being based upon the weight of said unleavened cookie composition.

7. A method as claimed in claim 1 wherein said heat treated composition is formed by heating said ingredients comprising flour and shortening or fat to at least 200° F. for at least 20 seconds.

8. A method as claimed in claim 1 comprising admixing a source of water with the heat treated cookie composition at a heat treated cookie composition temperature of about 100° F. to about 300° F. in an amount sufficient to form a dough-like mixture and wherein the total water content of said dough-like mixture is less than about 20% by weight, based upon the weight of

the dough-like mixture and said mixture has a water activity of less than about 0.7.

9. A method as claimed in claim 8 wherein the temperature of said heat treated composition is less than about 212° F. prior to said addition of the water.

10. A method as claimed in claim 8 wherein at least a portion of said sugar is admixed with the heat treated composition prior to said addition of the water.

11. A method as claimed in claim 1 wherein at least a portion of said sugar is subjected to said heating for forming said heat treated composition.

12. The method of claim 1 wherein said composition further includes an effective amount of at least one chemical leavening agent selected from the group consisting of sodium bicarbonate, sodium acid pyrophosphate, calcium carbonate, ammonium bicarbonate, and tartaric acid.

13. The method of claim 1 further comprising the step of shaping the unleavened cookie composition into cookie preforms at a temperature of about 100° F. to 150° F.

14. The method of claim 1 further comprising admixing at least one heat sensitive additive with the heat treated cookie composition at a temperature sufficiently low to substantially inhibit deactivation of the heat sensitive additive.

15. The method of claim 14 wherein said heat sensitive additive is selected from the group consisting of flavors, colors, fragrances, encapsulated additives, non-nutritive sweeteners, vitamins, and minerals.

16. The method of claim 14 wherein said heat sensitive additive is included in an amount of up to about 15% by weight based on the weight of the heat treated cookie composition.

17. The method of claim 14 wherein said heat treated cookie composition is cooled at least 35° F. before said heat sensitive additive is admixed.

18. The method of claim 1 further comprising admixing at least one shear sensitive additive with the heat treated cookie composition under conditions to substantially inhibit destruction of the shear sensitive additive.

19. The method of claim 18 wherein said shear sensitive additive is an edible particulate selected from the group consisting of candies, chocolate, raisins, dried fruits, and nuts.

20. The method of claim 18 wherein said unleavened cookie composition contains from about 5% to about 30% by weight of the shear sensitive additives based on the weight of the cookie composition.

21. A shelf stable cookie composition prepared according to claim 1 wherein said composition has a water activity of less than about 0.7 and is substantially unleavened and leavenable to a crumb-like structure by heating.

22. A method for the production of a shelf stable, leavenable cookie composition comprising:

- (a) admixing ingredients comprising flour and shortening or fat in a cooker extruder,
- (b) heating said ingredients in the extruder to a temperature of at least 150° F. to promote browning and to form a heat treated mass,
- (c) transferring the heat treated mass to a post extrusion mixing means and reducing the temperature of said heat treated mass,
- (d) admixing water with the heat treated mass at a heat treated mass temperature of about 100° F. to about 300° F. to obtain a substantially homogeneous unleavened cookie composition, and

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