

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO	
RAD: 13-295629- -5	FECHA: 2015-05-16 23:31:43
DEP: 2020 DIRECCION DE NUEVAS CREACIONES	EVE: 378 FASE NACIONAL INCLUIDO CAPITULO I
TRA: 11 PCT-PATENTE DE INVENCION CAPITULOS I Y II	FOLIOS: 9
ACT: 430 REQUERSOLICITA	

DIRECCIÓN DE NUEVAS CREACIONES

2020
Bogotá, D.C.,

Abogado(a)/Señor(a)
ALICIA LLOREDA RICAURTE

Asunto: Radicación: 13-295629- -5
 Trámite: 11
 Evento: 378
 Actuación: 430
 Oficio: 5370
 Folios: 9

Patente de Invención	X			Patente de Modelo de Utilidad	
Vía Nacional					
Vía PCT (Fase Nacional)	X	Capítulo I	X	Capítulo II	
No. Solicitud Internacional	PCT/EP2012/060193				
Fecha de Presentación Internacional	30/05/2012				

Como resultado del examen de fondo realizado, con fundamento en el Artículo 45 de la Decisión 486 de la Comisión de la Comunidad Andina, se comunica:

1. Documentos estudiados

Descripción:	Rad. N° 13-295629-00000-0000	18/Diciembre/2013
Reivindicaciones:	Rad. N° 13-295629-00004-0000	20/Noviembre/2014
Dibujos:	Rad. N° 13-295629-00000-0000	18/Diciembre/2013
Prioridad:	EP 11170231.2	16/Junio/2011

2. Análisis de la Prioridad

Se acepta la reivindicación de prioridad porque ésta solicitud fue presentada dentro del plazo establecido (Art. 9 D 486) y su contenido técnico corresponde con el de la prioridad reivindicada.

3. Objeto de la invención

De acuerdo con lo establecido en el numeral 1.2.2.2 del Capítulo Primero del Título X de la Circular Única para efectos del examen de patentabilidad, se tendrá en cuenta la tasa pagada por el solicitante al momento de comenzar el examen para las reivindicaciones adicionales; esto es que cuando la solicitud contenga más de diez (10) reivindicaciones y no se haya pagado la tasa complementaria, sólo se estudiarán las cubiertas por la tasa pagada. Por lo tanto, se estudiaran las reivindicaciones 1 a 10.

Título de la solicitud: “Proceso de tableta de caldo” (Ver página 1 del radicado N° 13-295629-00000-0000).

El problema técnico consiste en mejorar un proceso para la fabricación de caldos duros que tienen una o más capas de elementos visualmente atractivos, lo que permite un mejor control del proceso a través de una dosificación precisa, una manipulación suave y tratamiento de partículas secas, tales como rozos de verduras y hiervas.

Para resolver este problema técnico se presenta un procedimiento para preparar una tableta de caldo que tiene dos o más capas caracterizado por que comprende las etapas de formar una primera capa de tableta, formar una segunda capa de la tableta mediante la adición a la primera capa y comprimir las dos capas para formar la tableta.

El objeto de la presente solicitud se relaciona con la Clasificación Internacional de Patentes (CIP): A23L 1/00 // 1/40.

4. De la solicitud de patente

Titulo

El título no permite distinguir el objeto a proteger, por lo tanto es necesario cambiar el título, siendo este coincidente con el preámbulo de las reivindicaciones, por ejemplo el título podría ser “Un procedimiento para preparar una tableta de caldo que tiene dos o más capas”.

Reivindicaciones (Art 30 D 486, Art 22 PCT)

Durante la evaluación de claridad y concisión de las reivindicaciones, esta Oficina encontró que:

- La reivindicación 1 tiene por objeto un procedimiento, aunque muestra la secuencia de etapas técnicas por medio de las cuales se transforme una materia en un producto o se obtenga un resultado, sin embargo, los “pasos” se limitan a suministrar una serie de elementos (materias primas, equipos, etc.) pero no enseña las etapas técnicas con sus condiciones técnicas (temperaturas, flujos, presiones, etc.), por todo lo anterior esta reivindicación y sus dependientes no son claras. Se sugiere realizar las aclaraciones respectivas o definir la invención por sus características esenciales y estructurales, teniendo en cuenta de no modificar o ampliar la material ya divulgada.
- Las reivindicaciones 1, 4 y 8 a 11 no son claras porque presentan términos indefinidos y/o imprecisos “*mínimo*”, “*una o más*”, “*dos o más*” “*por debajo*”, “*inferior*” utilizados para definir el rango de varias características del procedimiento deben eliminarse. Estos términos no permiten establecer un intervalo puntual para cada una de las características técnicas, no siendo estas precisas y por lo tanto no claras, ya que no permiten una comparación con el estado de la técnica. Se sugiere sustituirlas por un término preciso o un rango concreto.
- Las reivindicaciones 1, 2 y 8 a 10 no son claras porque presentan términos generales “*potenciador de sabor*”, “*hiervas*”, “*aderezos*”, “*trozos de verduras*”, “*especias*”, “*grasa*”, “*aceite*”, “*fuerza de compresión*”, “*fuerza de compresión aplicada*”, “*formar una primera capa*”, “*formar una segunda capa*”, que sugieren de forma imprecisa que la protección se extiende a otras posibles variaciones o modificaciones. Se sugiere sustituirlas por un término preciso o un rango concreto.
- La reivindicación 1, referida a un procedimiento se incluyen características funcionales “*para formar la tableta*”, no se tendrán en cuenta para el estudio ya que dichas características si bien se pueden incluir para brindar claridad al objeto a

proteger, no le pueden dar novedad ni nivel inventivo a la invención. Las reivindicaciones deben caracterizar el procedimiento por una secuencia de etapas técnicas con sus condiciones (materias primas, equipos, temperaturas, flujos, presiones, etc.), por medio de las cuales se transforme una materia en un producto o se obtenga un resultado.

Descripción (Art 28 D 486)

Durante la evaluación de claridad y suficiencia de la descripción, esta Oficina encontró que:

- La descripción no muestra mediante análisis o ejemplos un efecto técnico sorprendente que evidencie el aporte de la invención al estado de la técnica.

5. Determinación del Estado de la Técnica

El estado de la técnica se determinó con base en la fecha de presentación de la solicitud de prioridad, es decir, 16 de junio de 2011.

Consultadas las fuentes de información con que se cuenta, se encontraron los siguientes documentos que anticipan el objeto de esta solicitud:

Ítem	Documento	Título	Fecha de publicación
D2	US 2007/0134397	“Solid concentrate for broths, bouillons, soups, sauces, (side) dishes, garnishing or seasoning and process for preparing the same”	14/Junio/2007
D3	US 5895675	“Production of powder-form and tablet-form salt- and fat-containing food products”	20/Abril/1999

El documento D2¹ divulga concentrados para caldos, caldos, sopas, salsas, salsas, platos, guarniciones, o para su uso como guarnición o condimento que contiene hierbas y / o legumbres secas, que se concentran es en la forma de un cuerpo sólido o de los órganos que tiene un peso de 2- 250 g, en particular, un cubo, bloque, o la forma de la tableta, lo que dio una sopa, salsa etcétera tras la dilución con agua (caliente) o un líquido acuoso, o puede ser utilizado como un (lado) del plato, el condimento o guarnición, y en donde las hierbas y/o legumbres secas están presentes en o con el concentrado como uno o más comprimidos partículas o capas (Resumen).

El documento D3² divulga un proceso para producir un polvo, producto alimenticio que contiene grasa. Al menos uno ingrediente cristalino de alimentos, tales como sal, y una grasa se mezclan para proporcionar una pasta. La pasta se muele luego para reducir el tamaño de los cristales del ingrediente alimentario cristalino y para recubrir los cristales con la grasa. La molienda se continúa hasta que se forma un polvo fluido. Si se desea, el polvo capaz de fluir luego puede ser presionado en comprimidos (Resumen).

6. Examen de Patentabilidad (Art 14 D486)

El siguiente examen de patentabilidad se realizó teniendo en cuenta las objeciones señaladas en el numeral 4 de la presente comunicación.

1 http://worldwide.espacenet.com/publicationDetails/originalDocument?CC=US&NR=2007134397A1&KC=A1&FT=D&ND=&date=20070614&DB=&&locale=en_EP
2 http://worldwide.espacenet.com/publicationDetails/originalDocument?CC=US&NR=5895675A&KC=A&FT=D&ND=&date=19990420&DB=&&locale=en_EP

Nivel inventivo (Art18 D486)

La reivindicación 1 consiste en “*Un procedimiento para preparar una tableta de caldo que tiene dos o más capas (...)*” (incluida bajo el radicado N° 13-295629-00004-0000).

Comparación de las características técnicas esenciales, con las del estado de la técnica:

Características esenciales	D2
Un procedimiento para preparar una tableta de caldo que tiene dos o más capas, caracterizadas porque comprende las etapas de:	Procedimiento para preparar un concentrado de comida, que comprende una o más partículas comprimidas o capas (Resumen; Reivindicación 3).
a) Formar una primera capa de la tableta que comprende una mezcla de mínimo dos de sal, potenciador de sabor, azúcar y grasa (o aceite)	20-98% (preferiblemente 40-95%, más preferiblemente 60-95%, todos los% en peso sobre el total de concentrado) ingredientes a granel que comprenden uno o más de sal, potenciadores del sabor, azúcar (s), grasa, aceite, gomas, espesantes, cremas, especias, almidón, almidón-derivados, harina, maltodextrina, hierbas de tierra, vegetales terrestres, legumbres tierra, extractos de carne, extractos vegetales (...) (B) la formación de 2-250 g (preferiblemente 3-50 g) de una mezcla de los ingredientes a granel a una partícula o capa, (Reivindicación 3). La formación en (b) puede ser por ejemplo, por prensado, moldeo o extrusión. Presionando y de extrusión se prefieren técnicas de formación a este respecto, presionando el más preferido (Párr. 38, Reivindicación 6).
b) Formar una segunda capa de la tableta mediante la adición a la primera capa de una mezcla que comprende una o más de hierbas, aderezos, trozos de verduras, y especias; y	2-80% (preferiblemente 5-60%, más preferiblemente 540%, todos en% en peso de concentrado total) de la hierba y / o trozos de verduras (...) A) prensar dicha hierba y/o trozos de verduras (preferiblemente 0,5-100 g de la misma) que tiene un contenido de humedad de 5-30% en peso de uno o más comprimidos partículas o capas (...) (Reivindicación 3).
c) Comprimir las dos capas para formar la tableta y en donde la tableta se forma usando una prensa rotativa de doble troquel.	Adherir 1-10 de las partículas o capas como se obtienen bajo (a) a 1-10 partículas o capas como se ha obtenido en (b), para obtener un concentrado sólido (artículo) comprimido (Reivindicación 3). El prensado u otra formación (tanto de hierbas / verduras, así como los ingredientes a granel o estos dos conjuntamente) se puede hacer uso de equipos como se conoce en la técnica para (comida) apremiantes o forman de material granular, por ejemplo, caldo de cubitos. Tal formación se puede hacer usando compactación de rodillo, extrusión, laminado, fundición, peletización, etc. (Párr. 55).
usando una prensa rotativa de doble troquel	No menciona.

El documento D2 se considera el estado de la técnica cercano a la invención definida en la reivindicación 1 porque divulga procedimientos para la fabricación de concentrados de comprimidos en capas.

La diferencia entre la invención definida en la reivindicación 1 y el procedimiento de D2 consiste en que en este último no se menciona el uso de una prensa rotativa de doble troquel.

No se evidencia un efecto técnico inesperado o sorprendente el hecho de que la prensa incluida sea una prensa rotativa de doble troquel, por cuanto el documento D2 divulga que el prensado u otra formación (tanto de hierbas/verduras, así como los ingredientes

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a granel o estos dos conjuntamente) se puede realizar haciendo uso de equipos conocidos en el estado de la técnica para la prensado y formación de productos (alimentos) de material granular, por ejemplo, cubos de caldo. Tal formación se puede hacer usando rodillos de compactación, extrusión, laminado, fundición, peletización, etc. (Párr. 55), obteniendo un producto sólido rígido en capas y estable (Ejemplo), por lo cual, el efecto sería el mismo.

Por lo tanto, el Problema Técnico Objetivo que pretende resolver esta invención se puede formular así: ¿cómo modificar el procedimiento conocido en D2 para tener otra alternativa en la obtención de una tableta de caldo de dos o más capas?

Sin embargo, una persona normalmente versada en la materia con el saber general de la técnica (*“procesos de aglomeración en la industria alimentaria: utilización de prensa rotativa de doble troquel para la fabricación de tabletas comprimidas (Fig. 7 (j))”*³) y con una serie de conocimientos específicos de la invención, estaría motivada a realizar experimentación de rutina y seleccionar una prensa rotativa de doble troquel para obtener el producto deseado en el procedimiento que se da a conocer en D2, para una dosificación precisa, distribución uniforme y una manipulación suave de los componentes en la obtención de una tableta de caldo de dos o más componentes, tal como se encuentra reclamado en la reivindicación 1 de la solicitud, motivo por el cual se considera obvia. Cabe decir, que la solución al anterior problema técnico objetivo se encuentra dentro de un diseño y optimización de procesos⁴, lo cual se encuentra dentro de la práctica habitual de la persona versada en la materia que trabaje en la formulación y fabricación de productos alimenticios.

Asimismo, el documento D2 divulga las características de las reivindicaciones dependientes así:

- Reivindicación 2, los concentrados son hechos de una capa soluble al agua o componentes fundibles en primer lugar, luego dicha capa es contactada con hierbas secas o partículas de vegetales, opcionalmente comprimidas, después de lo cual los vegetales del artículo son recubiertos con grasa fundida (Párr. 18).
- Reivindicación 3, el concentrado comprende aceite de palma (Ejemplos).
- Reivindicación 7, la capa que comprende los pedazos de vegetales comprende entre el 5 – 60% en peso del concentrado total (Reivindicación 1).
- Reivindicación 8, es posible de usar vegetales y hierbas secas, las cuales usualmente tienen un contenido de humedad menor al 5%, en particular 2% o menos (Párr. 46).
- Reivindicaciones 9 y 10, la compactación es usada por una compactación de rodillo, extrusión, laminado, colada, peletización, presión etc. (Párr. 0055). Los caldos duros, combinados en el caldo objeto contienen la composición de cada uno de las mezclas de caldos corresponden a la receta convencional de los caldos, que comprende sal, rellenos (maltodextrinas para mejorar la cohesión, almidón, maíz, almidón de papa,
- papa), grasa animal o vegetal, ingredientes de sabor como concentrados de sabores picantes o especias (Reivindicación 5; Párr. 25). Una de las capas es formada por partículas de plantas, en particular copos de hierbas, especias y pedazos de vegetales (Reivindicación 8).

³ Palzer et al. Agglomeration of pharmaceutical, detergent, chemical and food powders - Similarities and differences of materials and processes. Power Technology. Vol. 206, No. 1-2. 18 de enero de 2011. Pág. 2-17.

<http://www.sciencedirect.com/science/article/pii/S0032591010002445>

⁴ Perry Manual del Ingeniero Químico. Tomo I. Sección 2. Sexta edición. 1992. Págs. 2-81 a 2-85. Expediente 13-295629-5 2do Art. 45

Ahora bien, las reivindicaciones 4, 5 y 6 consisten en “*Un procedimiento de acuerdo con cualquiera de las reivindicaciones 1 a 3 (...)*” (incluida bajo el radicado N° 13-295629-00004-0000).

Comparación de las características técnicas esenciales, con las del estado de la técnica:

Características esenciales	D2	D3
Un procedimiento de acuerdo con cualquiera de las reivindicaciones 1 a 3, caracterizado porque:	Procedimiento para preparar un concentrado de comida, que comprende una o más partículas comprimidas o capas (Resumen; Reivindicación 3).	Un proceso para producir un producto alimenticio, como caldos, espesantes, sopas y salsa (Col. 1, líneas 5 a 8)
La fuerza de compresión aplicada para formar la primera capa es inferior a 40%, entre 15% y 35% de la fuerza de compresión aplicada para formar la tableta en el paso c)	No menciona.	Comprimir por presión el producto en polvo en forma de tabletas (Reivindicación 5).
La fuerza de compresión aplicada para formar la primera capa es de 0,1 a 1,5 kN, de 0,5 a 1,0 kN	No menciona.	La mezcla es puesta en una prensa y presionada para formar una tableta. La fuerza requerida para lograr una tableta con un grosor es determinada (Col. 5, líneas 66 a 67 a Col. 6, línea 1). La fuerza requerida para lograr una tableta de un grosor de 9.73 es de 1kN y de un grosor de 8.81mm es de 5kN (Ejemplo 2).
La fuerza de compresión aplicada para formar la tableta en el paso c) es 0,8 a 6kN, de 4 a 5 kN.	No menciona.	

Los documentos D2 y D3 se consideran el estado de la técnica cercano a la invención definida en las reivindicaciones 4, 5 y 6 porque divulgan procedimientos para la fabricación de concentrados de comprimidos en capas como tabletas de caldo.

La diferencia entre la invención definida en las reivindicaciones 4, 5 y 6 y el procedimiento de D2 consiste en que en este último no se menciona la magnitud de la fuerza de compresión aplicada durante las etapas de formación de la tableta de caldo.

El efecto que logra aplicar una fuerza de compresión menor para formar la primera capa en relación a la fuerza de compresión aplicada a las dos capas para formar la tableta de caldo, es que se proporciona una pre-compactación de los componentes de la primera capa, lo cual, facilita la operación y control del proceso de fabricación de la tableta de caldo, además de una manipulación suave de los componentes para hacerlo más eficiente.

Por lo tanto, el Problema Técnico Objetivo que pretende resolver esta invención se puede formular así: ¿cómo modificar el procedimiento conocido en D2 para facilitar la manipulación suave de los componentes, la operación y control del proceso en la obtención de una tableta de caldo de dos o más capas?

Sin embargo, la aplicación de una fuerza de compresión menor para formar la primera capa en relación a la fuerza de compresión aplicada a las dos capas para formar la tableta de caldo para facilitar la operación y control del proceso de obtención de la tableta de caldo, ya está divulgada en el documento D3 que se refiere a un proceso para producir un producto alimenticio, como caldos, espesantes, sopas y salsa (Col. 1, líneas 5 a 8), en el cual se comprime por presión el producto en polvo en forma de tabletas (Reivindicación 5), donde, la mezcla es puesta en una prensa y presionada para formar una tableta. La fuerza requerida para lograr una tableta con un grosor es determinada (Col. 5, líneas 66 a 67 a Col. 6, línea 1). La fuerza requerida para lograr una tableta de un grosor de 9.73 es de 1kN y de un grosor de 8.81mm es de 5kN (Ej. 2).

En consecuencia, la persona del oficio normalmente versada en la materia aplicaría una fuerza de compresión menor para formar la primera capa en relación a la fuerza de compresión aplicada a las dos capas para formar la tableta de caldo de D3 en el procedimiento de D2 para llegar así al objeto de las reivindicaciones 4, 5 y 6 de la solicitud. Por lo cual se consideran obvias. Adicionalmente, dado que el documento D3 enseña que la fuerza requerida para lograr una tableta de un grosor de 9.73 es de 1kN y de un grosor de 8.81mm es de 5kN (Ej. 2), una persona versada en la materia estaría motivada a realizar experimentación de rutina para determinar el rango la fuerza de compresión adecuado para aplicar durante la formación de la primera capa y el rango de fuerza de compresión aplicada a las dos capas para formar la tableta de caldo para obtener la tableta de caldo con el efecto deseado. Cabe decir, que la solución al anterior problema técnico objetivo se encuentra dentro de un diseño y optimización de procesos⁵, lo cual se encuentra dentro de la práctica habitual de la persona versada en la materia que trabaje en la formulación y fabricación de productos alimenticios.

Por tanto, las reivindicaciones dependientes 2 a 10 no mencionan características inventivas adicionales tampoco se pueden considerar inventivas.

Los requisitos de Patentabilidad de la presente solicitud están afectados así:

Item	Documento	Reivindicaciones afectadas	Requisito que afecta
D1	US 2007/0134397	1 a 10	Nivel Inventivo
D2	US 5895675	4, 5 y 6	Nivel inventivo

7. Respuesta a los argumentos del señor solicitante

Argumentos con respecto a D1

1. El señor solicitante argumenta que “... *el documento D1 no enseña ni sugiere un procedimiento incluya cada una de las características antes señaladas, principalmente **en dicho documento D1 de ninguna manera se revela o sugiere comprimir las dos capas para formar la tableta y en donde la tableta se forma usando una prensa rotativa de doble troquel** (...)*”

Se aclara que tiene razón, sin embargo, dicho documento tiene relación suficiente con el problema técnico que trata la invención “obtención de una tableta de caldo de dos capas estable”, por lo cual, afecta la altura inventiva de la solicitud, pero resulta más cercano el documento D2, por lo que se mantiene dicha anterioridad que afecta la solicitud por nivel inventivo.

Argumentos con respecto a D2

1. El señor solicitante sostiene “... *el documento D2 de ninguna manera puede anticipar el procedimiento reclamado actualmente, máxime si se tienen en cuenta que el procedimiento de D2, no divulga en particular la etapa de la compresión de las dos capas para formar la tableta y en donde **la tableta se forma usando una prensa rotativa de doble troquel***”.

Se aclara que aunque no afecta la novedad de la solicitud, si se ve afecta por nivel inventivo, dado que el documento D2 divulga que el prensado u otra formación (tanto de hierbas/verduras, así como los ingredientes a granel o estos dos conjuntamente) se puede realizar haciendo uso de equipos conocidos en el estado de la técnica para la prensado y formación de productos (alimentos) de material granular, por ejemplo, cubos de caldo. Tal formación se puede hacer usando rodillos de compactación, extrusión, laminado, fundición, peletización, etc. (Párr. 55), obteniendo un producto sólido rígido en capas y estable (Ejemplo), por lo cual, dicho documento afecta por nivel

⁵ Perry Manual del Ingeniero Químico. Tomo I. Sección 2. Sexta edición. 1992. Págs. 2-81 a 2-85.
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inventivo la invención.

Argumentos con respecto al estudio de novedad y nivel inventivo.

El señor solicitante argumenta que *“... Consecuentemente resulta evidente que el procedimiento para preparar una tableta de caldo que tiene dos o más capas que es el objeto de la reivindicación 1 no puede de ninguna manera ser anticipados por las enseñanzas de los documentos D1 ni D2, y por lo tanto es innegable su carácter novedoso (...) es importante destacar que el efecto técnico de esta característica técnica esencial consiste en permitir mejor control del proceso a través de la dosificación exacta, un manejo cuidadoso y el tratamiento de partículas secas, tales como trozos de hierbas y verduras (...) además, las hiervas y trozos de verduras no se encuentran unidos con firmeza (...)”*.

Se aclara que, el documento D2 enseña un procedimiento para preparar una tableta de caldo que tiene dos o más capas (Resumen; Reivindicación 3), que comprende: a) Formar una primera capa de la tableta que comprende una mezcla de mínimo dos de sal, potenciador de sabor, azúcar y grasa (o aceite) (Reivindicaciones 3 y 6, Párr. 38), b) Formar una segunda capa de la tableta mediante la adición a la primera capa de una mezcla que comprende una o más de hierbas, aderezos, trozos de verduras, y especias ((Reivindicación 3); y c) comprimir las dos capas para formar la tableta y en donde la tableta se forma usando una prensa rotativa de doble troquel (Reivindicación 3), y aunque el documento D2 no mencione específicamente una prensa rotativa de doble troquel, dado que la invención no muestra un efecto técnico sorprendente que haya sido demostrado, la persona versada en la materia seleccionaría la prensa adecuada que brinde la compresión deseada, dosificación exacta y manejo cuidadoso de los componentes para obtener una tableta de caldo de una o más capas uniforme, estable y con las características organolépticas deseadas, por lo tanto, es posible llegar a la invención reivindicada.

Cabe decir, que el procedimiento se caracteriza de forma muy general, y si bien, se presenta por la secuencia de etapas técnicas por medio de las cuales se transforma una materia en un producto, los “pasos” se limitan a suministrar una serie de elementos (materias primas, etc.), pero no enseña las etapas técnicas con sus condiciones técnicas (temperaturas, presiones, tiempos etc.), por medio de las cuales se transforme una materia en un producto o se obtenga un resultado; además, en el procedimiento se incluyen términos generales *“formar una primera capa”, “formar una segunda capa”*, características que no limitan el alcance de dicha reivindicación, por lo cual, no es claro el objeto que se busca proteger. Por tanto, la materia tal como fue reivindicada resulta obvia para una persona versada en la materia, y dado que persisten las objeciones que al respecto fueron trasladadas en el nuevo capítulo reivindicatorio, y que las diferencias mencionadas no se encuentran reflejadas en el capítulo reivindicatorio, esta Oficina considera que la invención carece de nivel inventivo.

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 (60) días contados a partir del día siguiente de la fecha de 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.

Se invita al solicitante se sirva indicar, si así lo considera, su renuncia al término faltante de sesenta (60) días para dar respuesta a este requerimiento y presentarla por escrito.

Finalmente, se le recuerda al solicitante que si como respuesta a este requerimiento llegara a modificar el capítulo reivindicatorio y éste contenga más de diez (10) reivindicaciones, no pagará la tasa de modificación voluntaria pero sí la correspondiente al pago por reivindicación adicional.

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.



JOSÉ LUIS SALAZAR LÓPEZ
Director de Nuevas Creaciones (c)

El anterior requerimiento fue notificado por fijación en lista, de fecha 2015-05-19

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Agglomeration of pharmaceutical, detergent, chemical and food powders – Similarities and differences of materials and processes

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ABSTRACT

Product developers tend to make a distinction between different types of agglomerated powders depending on whether they are produced by the pharmaceutical, detergent, chemical or food industry. This is perfectly valid for the hygiene, quality, safety or economical aspects of processes and products. However, from a process engineering point of view a different classification is needed in order to identify similarities between product categories and to facilitate an effective cross-fertilisation between different industries. The current contribution shows that a classification of the different materials according to their molecular polarity and their supra-molecular structure enables to understand the behaviour of the powder particles in different agglomeration processes. Basically one can distinguish between hydrophilic/hydrophobic amorphous and hydrophilic/hydrophobic crystalline materials. Depending on their polarity, particles made of amorphous materials can be plasticized using either polar or apolar liquids. They have a viscoelastic behaviour and their mechanical properties are strongly dependant on plasticiser content, temperature and deformation speed. Conversely, the mechanical properties of crystalline materials are hardly affected by the vapour pressure of plasticising liquids until they dissolve at a substance-specific vapour pressure value. The mechanical properties of crystals are also less sensitive to temperature changes. As a consequence, particles made of these four material classes have to be agglomerated differently which means different binder solutions and different process conditions are required. Moreover, the agglomeration processes applied by different industries are discussed while considering the mentioned product classes. Similarities between agglomeration technologies are elaborated and the reasons why certain processes are preferred by some industries are explained.

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1. Introduction

In various industries fine powders are produced in agglomerated form in order to improve their flowability, avoid lumping during rehydration, encapsulate sensitive components or simply to improve the optical appearance of the product. Most agglomerated powders are composed of polar molecules and are thus water-soluble. Some of these water-soluble substances are hardly affected by an increase in relative humidity, up to a critical value above which they dissolve at a substance-specific air humidity. Some others become increasingly sticky when exposed to an increasing relative humidity. Powders composed of apolar substances are water insoluble. Most of these powders are plasticized by apolar liquids and their viscosity decreases when increasing the temperature. A third class of powders is rather inert and hardly interacts with any liquids.

Increasing adhesion forces between single particles are a prerequisite for any agglomeration process. For industrial agglomeration of powders, increasing Van der Waals forces or material bridges between primary particles are relevant. Material bridges are generated

by sintering of amorphous materials or drying of liquid bridges containing dissolved crystalline substances. The processes which should be applied for increasing these forces strongly depend on the physico-chemical properties of the material building the particles.

By classifying the different powders according to their material properties and considering the process conditions applied during agglomeration similarities can be identified. In this context, we differentiate on one hand polar/apolar substances and on the other hand amorphous/crystalline supra-molecular structures. Furthermore one can distinguish between pressure agglomeration and agglomeration processes during which the primary particles collide more or less randomly.

2. Material properties of solids and adhesion forces between particles

2.1. Classification of solid materials according to their polarity and their supra-molecular structure

From a material science point of view, the solid materials building the particles can be classified independently of the involved manufacturing industry, but according to their interaction with polar

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liquids and according to their supra-molecular structure. Such classification enables to identify similarities between materials relevant to different industries.

Generally one can distinguish between polar and apolar substances.

Polar molecules contain mainly polar groups such as $-\text{OH}$, $-\text{COOH}$, $-\text{NH}_2$ and $-\text{SH}$ groups and occasionally short or medium chain alkyl groups, but no phenolic groups. They can thus interact with polar liquids such as water (a dipole), glycerol and ethanol. Most carbohydrates, organic and inorganic acids and their salts, and various mineral salts are polar substances that dissolve in aqueous solutions. Microcrystalline cellulose is an example of a polar non-dissolving solid that interacts with water.

Conversely apolar molecules consist of phenolic groups and medium/long chain alkyl chains. They interact primarily with apolar liquids such as chloroform, triacetin, carbon acids, carbon acid esters, phthalic acid and toluene. Typical examples for apolar substances are fats, waxes and plastics such as polystyrene, polypropylene and polyethylene.

Finally some solids are rather inert. Examples for such substances are aluminium oxide, different metals, silica, glass, carbon and lime stone. The majority of scientific studies are unfortunately mainly carried out with such inert particles, and thus the applicability of the results to industrial agglomeration processes remains questionable.

The nature of the interaction between solids and liquids strongly depends on their polarity and on the free volume within the supra-molecular solid structure. Small solvent molecules with similar polarity than the matrix can migrate into a molecular matrix, which is characterized by a large free volume. These small solvent molecules increase the mobility of the larger matrix molecules, and therefore they act as a plasticiser. Solvent molecules can hardly penetrate into molecular structures consisting of a small specific free volume. However such solid structures still might dissolve in liquids of similar polarity.

The free volume in a molecular assembly is linked to its degree of order. According to the molecular order one can distinguish between amorphous systems (e.g. glass) in which the molecules are arranged more or less statistically, semi-crystalline and crystalline structures in which the atoms or molecules are highly ordered. At a given temperature, the free volume in amorphous matrices is generally higher than that in equivalent crystalline structures.

Some crystalline matrices are built of a continuous network of molecules/atoms which has no clearly defined boundaries or single geometrically clearly shaped crystals. For example, carbon and metals (e.g. iron) crystallize forming such networks. Conversely, ionic solids such as sodium chloride and calcium carbonate form single, clearly defined crystals. Also low molecular weight substances (e.g. water or dextrose) can crystallize forming defined geometrical structures. Substances characterized by a large molecular weight can form tri-dimensional crystalline regions build of polymer chains, which are orientated in parallel (e.g. amylopectin or PET).

Amorphous polymers are characterized by a statistical conformation of polymer chains, which is referred to as random coil. Because these random coils can be elongated while the system is exposed to stress, amorphous polymers have viscoelastic properties.

The crystalline and the amorphous state are characterized by different optical, mechanical and thermodynamic properties. These properties are used to determine the degree of crystallinity of powders.

In principle, all substances can exist in the crystalline, semi-crystalline or amorphous state. For example, low molecular weight organic acids (e.g. citric acid) or mono- and disaccharides (e.g. lactose and sucrose) are found in the amorphous and crystalline forms and different crystal morphologies can be distinguished.

The state of a specific substance depends on the mobility of the molecules as well as the velocity at which the liquid/melt is transformed into a solid. Fast cooling or rapid removal of solvent generally leads to amorphous structures. Upon drying or cooling, the

viscosity rises leading to the formation of first a thick syrup and eventually an amorphous glassy solid. Amorphous structures can also be formed when grinding crystalline low molecular substances (Roth 1976; Niediek and Barbenics 1980). For slow processes, the molecules have enough time to arrange themselves into a highly ordered crystalline structure. During annealing, the crystallinity might increase significantly [11].

As mentioned before, the free volume v_f determines the interactions between the solid and the small molecules. It also gives information on the motion of the molecules and can thus be used to determine the molecular mobility. The free volume is equal to the non-occupied space in a molecular matrix. If a critical free volume is accumulated near a molecule, the latter can change position. The free volume v_f is equal to the difference between the total specific volume v , and the occupied volume v_o (see Eq. (1)).

$$v_f = v - v_o \quad (1)$$

The free volume can be measured by Positron–Annihilation–Lifetime-Spectroscopy (PALS). This technique is based on the dependency of the lifetime of a positron with the electron density of the investigated medium [12,29,41].

The free non-occupied volume v_f is obtained by integrating the product of the number density distribution $N_h(r)$ and the corresponding hole volume $v_{h(r)}$ [29] (see Eq. (2)).

$$v_f = \int N_h(r) \cdot v_{h(r)} dr \quad (2)$$

r is the hole radius.

Various authors, amongst them [22], have shown that below and above the melting or glass transition temperature, the fractional free volume f increases linearly with temperature (see Eq. (3)).

$$f = \frac{v_f}{v} = f_0 + \alpha_f \cdot (T - T_0) \quad (3)$$

T is the temperature, T_0 represents the reference temperature, f_0 is the fractional free volume at T_0 and α_f is a coefficient.

When heating a crystalline system, its molecules increasingly vibrate around their position in the lattice and at a critical temperature (called the melting point) the crystalline structure breaks. At this temperature the free volume and the average hole radius also shows a discontinuity. The resulting melt can then re-crystallize if cooled slowly enough. When heating an amorphous glass, the free volume and the average hole volume increase steadily. However, above a certain temperature the gradient with which both volumes increase changes significantly. Molecule clusters are set free and start to rotate and slip past each other. The lower border (onset) or the mean (midset) of this temperature interval is defined as the glass transition temperature T_g . The corresponding change in the state of the amorphous molecular matrix is called glass transition.

The free volume determines the viscosity of a molecular assembly. [13] assumed an exponential relation between the viscosity of a polymer η and its free volume v_f as described in Eq. (4).

$$\ln(\eta / \text{Pa} \cdot \text{s}) = \ln A + B^* \cdot (v - v_f) / v_f \quad (4)$$

A and B^* are dimensionless constants.

According to the different physical properties of amorphous and crystalline structures and the obviously different interaction of polar and apolar substances with solvents, it is essential to classify powders before discussing their agglomeration behaviour.

Fig. 1 illustrates the possibility to classify solid materials into 4 categories:

- Polar amorphous solids (e.g. carbohydrates and amorphous organic acids)

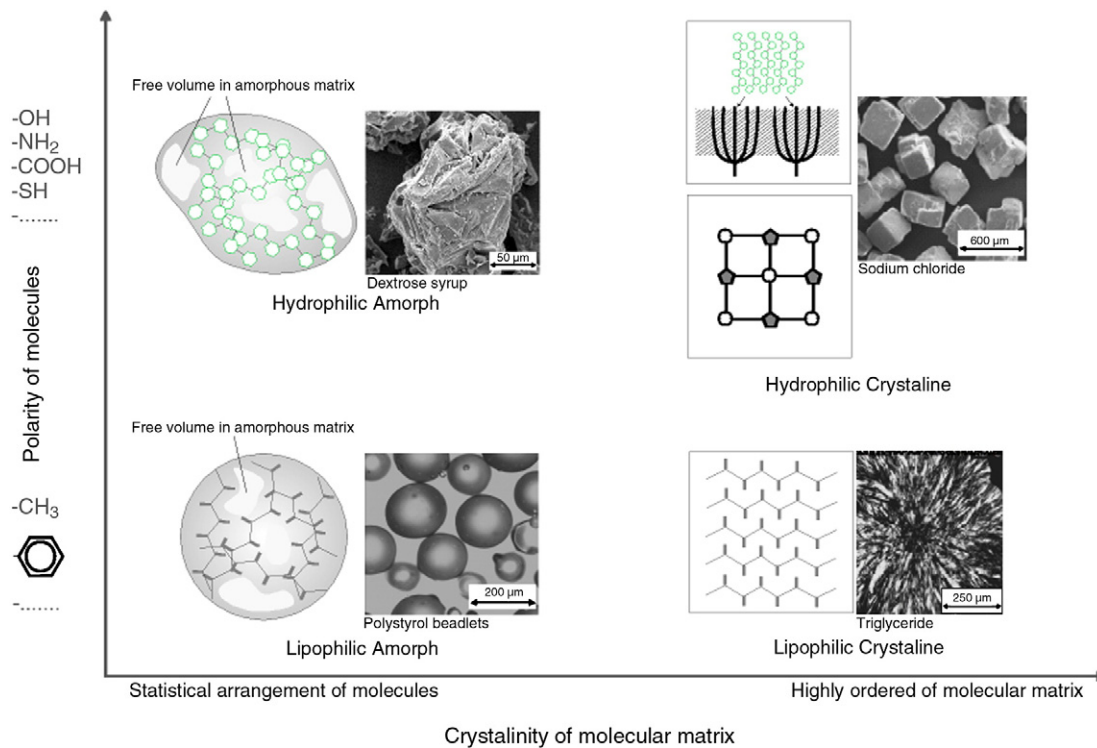


Fig. 1. Classification of solids according to their polarity and supra-molecular order.

- Apolar amorphous solids (e.g. polystyrene, polypropylene, and waxes)
- Polar crystalline solids (e.g. mineral salts, crystalline organic acids, and urea)
- Apolar crystalline solids (e.g. mono- and tri-glycerides)

This classification which is relevant for all types of powders enables to identify similarities between different base powders. In addition, it allows selecting the appropriate agglomeration technology for a given product.

For pressure agglomeration of amorphous polymers, the temperature dependency of their viscosity, sometimes also referred to as thermoplasticity, is relevant. In order to demonstrate that all amorphous polymers exhibit similar thermoplastic properties, the temperature dependency of the mean hole volume of three water-soluble carbohydrate polymers is compared with the one of the two water insoluble plastic polymers, polystyrene and polypropylene. As seen in Fig. 2, the average hole volume, which can be correlated with the viscosity [13], increases linearly with temperature. At the glass transition temperature of the different materials the gradient with which the average hole volume increases changes significantly. The absolute hole volume in the two plastic polymers is significantly higher than that in the dextrose polymers. This might be linked to the broader distribution of the molecular weight of the dextrose polymers, leading to shorter polymer molecules filling the holes present between the longer molecules. Conversely, the plastic polymers consist of a narrower molecular weight distribution. The average hole size in the dextrose polymer matrix is thus lower than in the plastic polymers. It is also noteworthy that in principle all amorphous polymers (whether water-soluble or insoluble) show a clear glass transition and behave more or less equally when the temperature increases. This therefore clearly demonstrates similarities between amorphous polymers used in different industries for manufacturing agglomerated products.

The average hole volume also depends on the plasticiser content. While amorphous solids absorb liquids of similar polarity, the average hole volume in the molecular matrix increases significantly [28].

2.2. Interaction of crystalline and amorphous solid materials with liquids

In the frame of various agglomeration processes applied by different industries, liquid binder solutions are added to the particles in order to build material bridges between them. The interaction between the material building the particles and the liquid is decisive for the stability of the formed bridges. Even for pressure agglomeration of amorphous particles their interaction with plasticising liquids

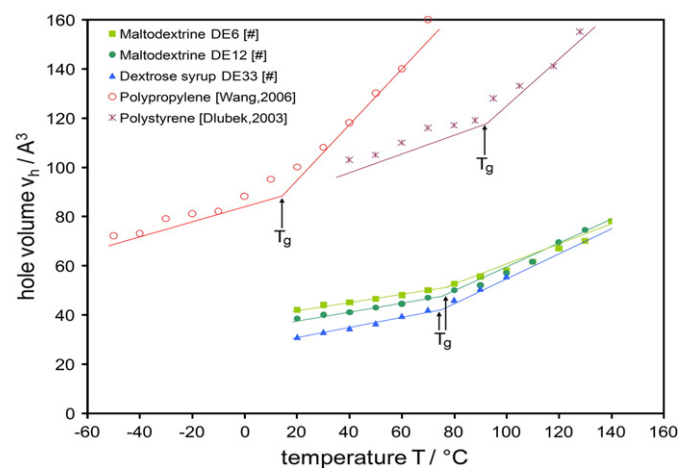


Fig. 2. Hole volume in amorphous maltodextrin or dextrose syrup (at $a_w = 0.22$; 5–8% water), polypropylene and polystyrene matrices measured by PALS depending on temperature #: (Kilburn et al., 2005 [29]; Wang et al., 2006 [60]; Dlubek et al., 2003 [12]).

is relevant. The viscosity of the amorphous material which determines the achieved deformation depends on the plasticiser content. Accordingly the interactions of solids with liquids have to be analyzed for the different material classes described above.

The amount of liquid absorbed by a solid substance depends on the liquids polarity. When it is not in direct contact with the solid, it also depends on the partial pressure of the liquid in the surrounding gas phase. Liquids are only absorbed by solids characterized by a similar polarity.

The water content of hydrophilic polar substances depends on the water activity or the relative humidity of the surrounding air. The sorption isotherm describes the relation between water content (w') and water activity a_w for a given temperature. It can be modelled according to Guggenheim, Anderson and deBoer (GAB equation) [61]

$$\frac{w'}{w_m} = \frac{C \cdot K \cdot a_w}{(1 - K \cdot a_w) \cdot (1 + (C-1) \cdot K \cdot a_w)} \quad (5)$$

C and K are two parameters and w'_m is the amount of water (on a dry basis) required for a monomolecular layer of water molecules on the solid surface. With $K=1$, the GAB equation can be simplified and is then referred to as Brunauer, Emmett and Teller equation or BET equation [9].

Due to a larger free volume within the molecular matrix, the water capacity of amorphous substances is larger than that of crystalline materials. In most crystalline matrices the free volume is not sufficient for the incorporation of significant water quantities. Consequently, below a critical relative humidity leading to dissolution of the crystalline substance, a significant quantity of water can only be stored in the crystalline matrix in the form of crystal water. Amorphous hydrophilic substances behave differently when they are exposed to an increasing relative humidity. They absorb increasing amounts of water when increasing the relative humidity. The water molecules are stored within the free volume v_f left within the amorphous matrix. The viscosity of the amorphous melt decreases to eventually a diluted solution of the hydrophilic substance is obtained. In contrast to crystalline materials, a critical humidity at which particles might dissolve can thus not be defined. Typical sorption isotherms of water-soluble amorphous and crystalline solids used in agglomeration of pharmaceutical, food and chemical powders are shown in Fig. 3. The amount of absorbed water appears to be comparable for most water-soluble amorphous solids and most water-soluble crystalline substances clearly dissolve at water activities above 60%.

Absorption of certain liquids by amorphous solids often changes the mechanical properties of the particles. Such liquids, called plasticisers, have a similar polarity than the solid and thus can penetrate into the amorphous matrix. According to [34] the fractional free volume at the glass transition point f_g is similar for all substances ($f_{gs} = f_{gw}$). Furthermore, the fractional volume of the moist product f is equal to the sum of the fractional volume of water f_w and the fractional volume of the solid f_s (see Eq. (6)).

$$\begin{aligned} f &= (1-w) \cdot f_s + w \cdot f_w & \alpha_f &= (1-w) \cdot \alpha_{fs} + w \cdot \alpha_{fw} & f_{gs} &= f_{gw} = f \\ \Rightarrow f &= (1-w) \cdot (f_{gs} + \alpha_{fs} \cdot (T - T_{gs})) + w \cdot (f_{gw} + \alpha_{fw} \cdot (T - T_{gw})) = f_g + \alpha_f \cdot (T - T_g) \\ \Leftrightarrow T_g &= \frac{(1-w) \cdot T_{gs} + w \cdot k \cdot T_{gw}}{(1-w) + w \cdot k \cdot \alpha_{fw}} & \text{with } k &= \frac{\alpha_{fw}}{\alpha_{fs}} \end{aligned} \quad (6)$$

$T_{g,s}$ is the glass transition temperature of the solid, $T_{g,w}$ represents the glass transition temperature of the pure liquid (e.g. water), w is the liquid content and k the Gordon & Taylor constant [23]. The plasticisation of dextrose syrup by polar liquids (water and glycerol) was compared with the plasticisation of polystyrene by apolar liquids

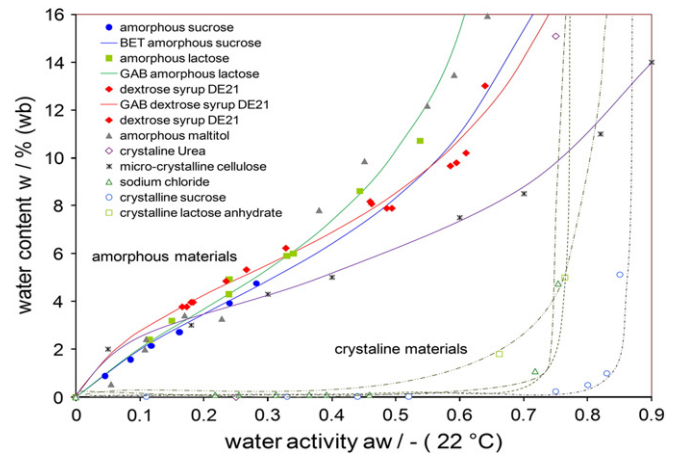


Fig. 3. Sorption isotherms at 22 °C of different hydrophilic amorphous and crystalline solids.

(phenylsalicylate, chloroform and methylsalicylate) (see Fig. 4). Obviously polar amorphous solids such as spray-dried dextrose syrup can be efficiently plasticized by the polar liquids glycerol and water. When increasing liquid content the glass transition temperature and the viscosity of the solid decreases. A similar effect is observed when adding apolar liquids to apolar amorphous solids. Independently on the considered application and substance class, amorphous solids can effectively be plasticised by liquids of similar polarity.

Hydrophilic amorphous and crystalline materials also differ in their dissolution behaviour when they are immersed into liquids of similar polarity. Amorphous water-soluble substances that are in direct contact with bulk water or aqueous solutions absorb the water molecules and the viscosity decreases steadily until the particles disintegrate into single molecules. Such dissolution process is rather exothermic due to high energetic level of the amorphous matrix [33]. Dissolution of crystals immersed in water occurs layer by layer. Nearly no water directly migrates into the crystalline structure. Because of the low energetic level of crystalline structures, the dissolution process is endothermic. Fig. 5a and b shows images of dissolving amorphous and crystalline particles. In various agglomeration processes liquid droplets are sprayed on moving amorphous particles. The droplets penetrate into the amorphous polymers and decrease locally their viscosity. Simultaneously polymer molecules might also migrate into the liquid droplets, generating consequently a viscous solution at the particle surface. If a second particle impact on this solution, then a viscous bridge is formed. Liquid droplets colliding on crystalline surfaces do not migrate into the solid matrix. Although molecules are dissolving within the liquid, its viscosity remains moderate because most of the crystallizing substances have a comparable low molecular weight.

2.3. Mechanical properties of particles to be agglomerated

The type of agglomeration process and the optimal process parameters depend on the mechanical properties of the solid material building the particles. The mechanical properties are characterized by the viscosity and elasticity or the storage and loss moduli of the material. The viscosity and elasticity of amorphous solids might vary as a function of the process conditions by several orders of magnitude.

Following the time-temperature superposition principle, all relaxation mechanisms exhibit the same temperature dependency [16,35,64]. According to this principle, viscoelastic data (like viscosity, compliance or modulus) obtained for one temperature and time or

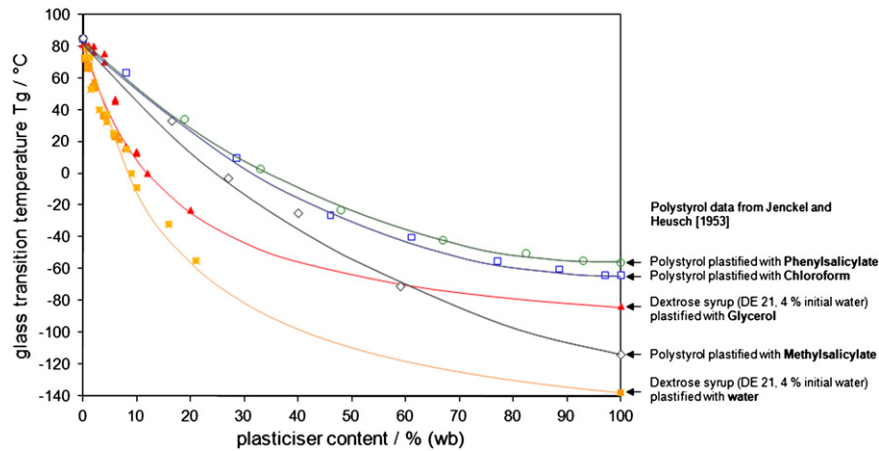


Fig. 4. Glass transition temperature of dextrose syrup and polystyrene as a function of the plasticiser content (Jenckel and Heusch, 1953 [25]).

frequency can be extrapolated to a different temperature by multiplying the logarithmic time or frequency values with a temperature-dependent shift factor a_T . [64] suggested the following equation for the viscosity shift factor:

$$a_T = \frac{\eta \cdot T_0 \cdot \rho_0}{\eta_0 \cdot T \cdot \rho} \quad (7)$$

η is the viscosity of the material, T represents the temperature and ρ is the density.

Combining Eqs. (3) and (4) with Eq. (7), the Williams, Landel and Ferry (WLF) equation is obtained. For deducing Eq. (8) Williams, Landel and Ferry (1955) assumed that the product of absolute temperature and density is roughly constant.

$$\log a_T = \frac{\eta}{\eta_0} = \frac{-\frac{B^*}{2.303 \cdot f_0} \cdot (T - T_0)}{\frac{f_0}{\alpha_f} + T - T_0} = \frac{-C \cdot (T - T_0)}{B + (T - T_0)} \quad (8)$$

T_0 is a reference temperature and C and B (in Kelvin) are assumed to be constant factors. C has a theoretical value of 17.

The viscosity of solids with high dry matter content can be measured by dynamic mechanical thermal analysis (DMTA). DMTA measurements deliver the storage and loss moduli of the material. Knowing the storage modulus G' and the loss modulus G'' the complex viscosity η^* can be calculated for each frequency ω according to Eq. (9) [21]:

$$\eta^*(\omega) = \frac{[G'^2 + G''^2]^{0.5}}{\omega} \quad (9)$$

Empirically, Cox and Merz (1958) found that steady state shear viscosity $\eta(\dot{\gamma})$ and complex viscosity $\eta^*(\omega)$ are numerically equal if the shear rate $\dot{\gamma}$ and the frequency ω are numerically identical.

A so-called viscosity master curve can be established to compare complex viscosities obtained for different temperature/frequency combinations. For each temperature/frequency combination the complex viscosity is calculated using the measured storage and loss moduli. The complex viscosity is divided by the zero shear viscosity corresponding to the temperature of the measurement. The frequency of the oscillating stress is multiplied with the characteristic relaxation time, which already includes the shift factor a_T . Finally a viscosity master curve for the complex viscosity η^* , can be produced by plotting the ratio η^*/η_0 against the product of frequency and characteristic relaxation time $\omega \cdot \tau^{\#}(T)$. By using the generated master curve the

complex viscosity for each frequency/temperature combination can be determined.

In order to point out similarities between different materials, the viscosity master curves of dextrose syrup is compared with the one of star structured and linear plastic polymers as published by Ngai [41]. The modulus data of the dextrose syrup were already reported in previous papers [42]. The result of this comparison is shown in Fig. 6. In principle the frequency and relaxation time dependency of all polymers are similar. Just the viscosity values for the dextrose syrup as measured by DMTA are at low frequencies one order of magnitude higher than the theory would suggest. This might be linked to the design of the DMTA trials for which the dextrose powder is packed into a plastic pouch.

According to these results the frequency and relaxation time dependency of the complex viscosity are similar for all investigated polymers. Since the relaxation time also includes the viscosity shift factor (which is a function of the temperature; see Eq. (8)) the temperature dependency of the viscosity is also similar for the investigated substances. The complex viscosity of the material determines the process conditions required for pressure agglomeration. The lower the complex viscosity the lower the pressure and compression time needed to achieve a significant deformation of the particles [42].

2.4. Adhesion forces generated during agglomeration

Increasing adhesion forces are a prerequisite for any agglomeration process. The adhesion forces strongly depend on the mechanical properties of the solid material building the particles.

In a number of agglomeration processes the particle surface is coated partially by liquid binder in order to generate liquid bridges between randomly colliding inert particles or crystals in which the liquid cannot migrate. The generated adhesion forces are capillary forces as described by Rumpf et al. [50,51]. These weak forces have to be increased by successive drying to generate stable agglomerates. In order to improve the strength of the moist liquid bridges, polymers (e.g. starch hydrolysates) can be dissolved in the liquid. Another possibility is to decrease the mean particle diameter. This significantly improves the stability of the moist ("green") agglomerates. If a liquid binder of a similar polarity than the solid is applied onto the surface of an amorphous particle, it migrates into the particle surface that it plasticises (see Fig. 4). If the viscosity at the particle surface decreases locally below 10^6 Pa s the particle surface becomes sticky. If two particles collide rapidly after the liquid droplet is placed on the solid surface of one of the two particles a liquid bridge is formed. The

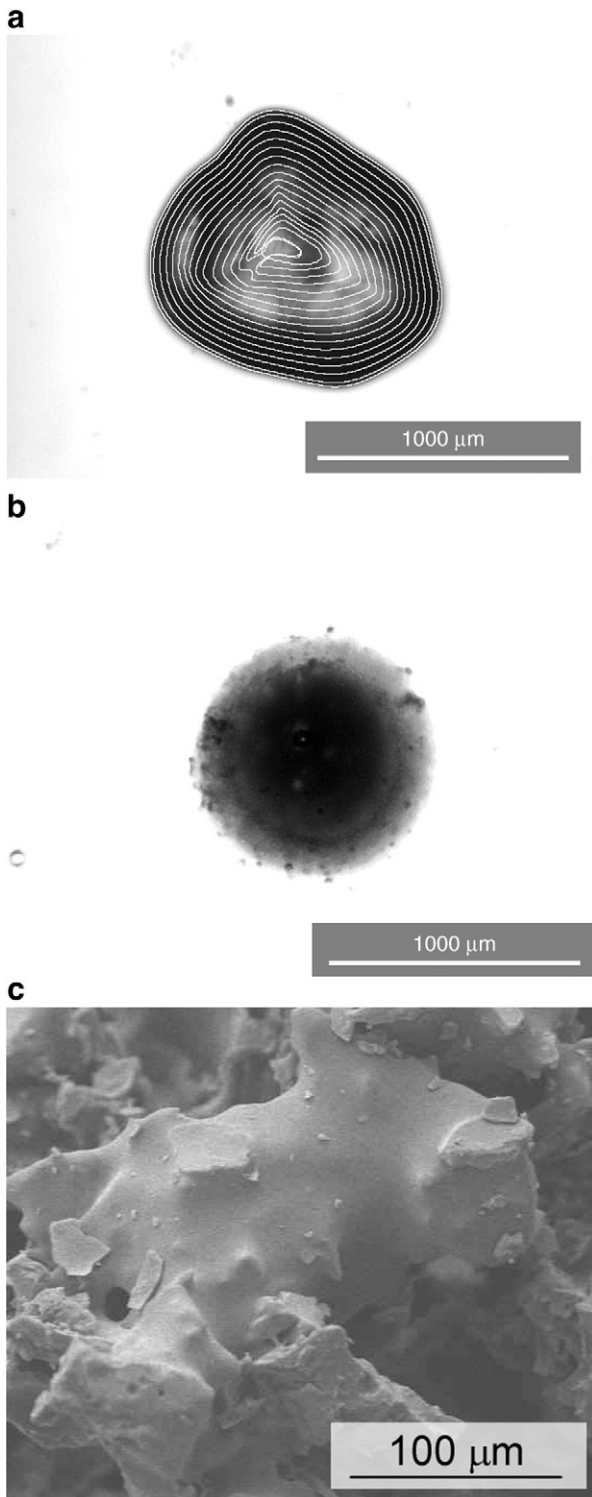


Fig. 5. Dissolution of water-soluble particles (25 °C, natural convection of water) and local plasticisation of an amorphous surface due to impacting droplets. a) Dissolving citric acid crystal (distance between lines corresponds to 5 s) [Marabi, 2007]. b) Dissolving amorphous maltodextrine particle [provided by Marabi, 2010]. c) Plasticised surface of maltodextrine particle.

strength of this bridge depends on the capillary forces. Amorphous substance dissolves in this bridge and thus the strength of the bridge increases. If the collision takes place after the liquid has migrated into the amorphous matrix, the establishment of a material bridge is governed by the viscosity. Sintering generates viscous bridges between plasticised amorphous particles. The stability of the

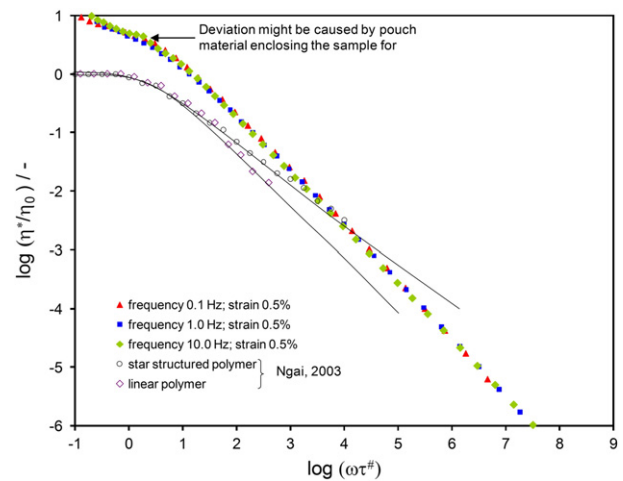


Fig. 6. Viscosity master curve of dextrose syrup ([42] & 2009b), a star structured plastic polymer and a linear plastic polymer according to Ngai [41] ($\eta_g = 10^{13}$ Pa s).

generated sinter bridge depends on its diameter (which is a function of the sinter time) and the viscosity of the material building the bridge. These considerations are relevant to all agglomeration processes in which particles are fluidized mechanically or pneumatically, independently on the considered industry. Particles collide randomly and liquid or sinter bridges are built between them. The adhesion forces are either capillary forces (liquid bridges) or viscous forces (sinter bridges).

Also increasing Van der Waals forces between particles can be used for agglomeration. During pressure agglomeration processes such as roller compaction or tableting particles are compressed to decrease the distance between their surfaces and to increase the contact area. Crystalline particles deform plastically. The Van der Waals forces achieved between such particles are a function of the maximal compression pressure applied and the yield stress of the material. Compressing viscoelastic amorphous polymers the strength of the Van der Waals forces depend on the complex viscosity of the material (which is a function of frequency, temperature and plasticiser content) and the compression force and the compression time [42]. In this sense tableting of plastic polymers is comparable to tableting of starch hydrolysates such as maltodextrine.

3. Agglomeration processes applied by different industries

Various processes are used by the industry for powder structuration: agglomeration during drying, fluid-bed agglomeration, steam-jet agglomeration and different pressure agglomeration processes (Pintauro, 1972; Jensen, 1975; [28,46,54]). Depending on to the powder properties different agglomeration technologies have to be applied to combine fine primary particles to larger ones. In this context we will only discuss processes used for the production of free flowing granules of a size range comprise between 100 and 5000 mm or for manufacturing larger tablets.

In order to point out similarities between agglomeration processes applied by different industries the different technologies are divided into different groups which are discussed in the context of the described material classes.

One can principally distinguish between processes during which sticky particles collide more or less randomly with each other, processes during which solid bridges are formed by drying or sintering between non-moving particles and processes during which a bulk of static powder particles is compressed into a compact.

During spray drying, spray chilling, steam-jet agglomeration, fluid-bed agglomeration, disk agglomeration and mixer agglomeration

particles collide randomly. During integrated belt-drying (a combination between spray and belt-drying), belt-drying and batch-wise oven drying particles having no relative velocity agglomerate together by drying of liquid bridges or sintering. Typical pressure agglomeration processes are tableting, roller compaction and extrusion of wet powder masses.

Table 1 shows a summary of the most important agglomeration processes used by the food, pharmaceutical, detergent and fine chemicals industry. Although equipment used by different industries may vary, the basic agglomeration principle remains the same (Fig. 7).

Spray drying, integrated belt-drying and steam-jet agglomeration are continuous processes. Depending on the batch sizes fluid-bed and mixer agglomeration are preformed either as batch or continuous process. Steam jet, disk agglomeration, extrusion, tableting and roller compaction are by their nature continuous processes.

Taking into account the introduced material classification the main agglomeration principles will be discussed. Similarities and differences in powder agglomeration in different industries are highlighted.

3.1. Agglomeration by random collision of sticky particles

During spray drying, spray chilling, steam jet agglomeration, fluid-bed agglomeration and mixer agglomeration, adhesive droplets or particles collide randomly. During spray drying and spray chilling, liquid droplets are transformed into a powder whereas during the other agglomeration processes dry particles are rewetted or partially coated with a binder liquid. The adhesion of the particles, which form low density agglomerates, depends on the surface viscosity, the contact angle of the liquid/solid system, the surface tension of the liquid at the particle surface, the contact time and the kinetic energy of the particles. Adhesion depends on all these parameters. Stickiness and adhesion are generally observed if the surface viscosity of the particles is below $10^8 \text{ Pa}\cdot\text{s}$ [31,42].

3.1.1. Agglomeration of detergent, chemical and pharmaceutical powders during spray drying

Various different food powders such as infant formulas, milk powders, soluble coffee, flavours, starch hydrolysates, pharmaceutical excipients, base powders of detergent formulations, zeolite powders used in detergents, fine chemicals and enzyme or micro-organism containing powders are spray-dried. All these powders are water-soluble and most of them are characterized by amorphous structures. To improve handling and dissolution properties, agglomeration of the primary particles generated by drying of single droplets is generally desired. Thus agglomeration, during spray drying is relevant for several industries.

During spray drying a concentrated solution is sprayed into drying towers where the droplets are dried by a counter- or co-current flow of hot air. The fast dehydration leads to the formation of amorphous structures. If small (and thus highly mobile) molecules are present, these structures are rapidly transformed into crystals. If the molecular mobility is low, the final powder particles tend to have an amorphous structure. The fine particles separated from the exhaust air are added back into the dryer and generate agglomerates [19,39,47,55].

Particles composed of amorphous water-soluble substances can be easily agglomerated during spray drying. Hoffmann et al. [24] and [6,7] discussed the stickiness of such amorphous water-soluble powders during spray drying. Adhikari et al. [1] and Werner et al. [62] investigated the stickiness of amorphous carbohydrate solutions with different water contents by using a tack tester installed in a kind of drying channel. Both authors identified a substance-specific humidity value providing a maximal pull-off force. At both low and high moisture content the stickiness was significantly reduced. Particles were adhesive if their surface viscosity was below $10^8 \text{ Pa}\cdot\text{s}$ (solid state). Low-viscous droplets with viscosities below $10^3 \text{ Pa}\cdot\text{s}$ (liquid-like state) simply coalesce with each other or coat larger dry particles. In both cases no

porous agglomerate structures are obtained. Decreasing the moisture content of droplets containing water-soluble oligo- or polymers, their stickiness increases until a maximum adhesion force between colliding droplets is reached. Under such conditions, colliding semi-dried droplets can form stable porous agglomerates due to viscoelastic deformation of their surface or rapid sintering of amorphous substances. Further drying increases the droplets viscosity. Subsequently, the diameter of the newly established sinter bridges as well as the achieved deformation of the droplet surface both decrease with progressing drying. At water contents corresponding to viscosities exceeding $10^8 \text{ Pa}\cdot\text{s}$ the energy provided by the collision is insufficient to achieve a significant deformation and the short contact time between the particles no longer enable the formation of sinter bridges.

However, it has to be considered that the state of maximal stickiness depends on the kinetic energy of the colliding particles and the contact time of the collision partners. The contact time is determined by the mechanical properties and the kinetic energy of the colliding particles [17]. Assuming that for amorphous solids sintering is the main adhesion mechanism the diameter of the established sinter bridges can be estimated through the known sinter equations. Measuring or modeling (CFD) the moisture and temperature distributions in the spray tower, the locations where the particles are adhesive (but not liquid) can be identified [20,44]. At such locations re-circulated dry fine particles should be added in order to agglomerate them with drying particles. If sticky particles collide with each other or with the added dry particles, they adhere to the collision partner forming porous agglomerates.

Amorphous primary particles are relatively spherical and agglomerates build of such units are characterized by an irregular form composed of spheres and spherical calottes (see Fig. 8a).

For the drying of solutions that contain water-soluble low molecular weight substances (e.g. mineral salts, see Fig. 8b), adhesion of the generated particles is more difficult because of their high molecular mobility and subsequent rapid crystallization. Even highly concentrated solutions of such substances have a low viscosity. Thus the stability of liquid bridges generated between particles is low and primary particles have to be small in order to form stable agglomerates. For efficient agglomeration small droplets and particle sizes are required and a saturated liquid solution must be present at the surface of the crystals built during drying. The formed agglomerates are often very brittle. The regular geometrical pattern of the crystals building the agglomerates can be easily identified, these rapidly formed crystals are often not characterized by sharp edges (see Fig. 8b). Depending on the molecular mobility and the dehydration time mixes of crystalline and amorphous structures might also be obtained during drying. The amorphous part is transformed into more stable crystals during post-drying and storage.

Agglomeration during spray drying is applied in the food, chemical and detergent industries. Independently on the application, industry and supra-molecular structure of the base powder, irregular shaped, highly porous and comparable brittle agglomerates are obtained (see Fig. 8). These agglomerates re-hydrate fairly quickly because of their irregular structure and high porosity.

A spray drying process particularly suitable for drying and agglomeration of sticky or high fat powders was developed by combining spray- and belt-drying (see Fig. 7b). In an integrated belt-drying the liquid to be dried is atomised into a short spray tower through a rotary atomiser or a set of pressure nozzles. Particles that might be still in their sticky state fall on a moving perforated polyester belt where they agglomerate, thus forming a powder cake. This powder cake is dried by a vertical downwards-oriented stream of hot air while carried by the belt to the cooling zone [3,36].

3.1.2. Steam-jet agglomeration of food powders

Different powders are agglomerated by steam-jet agglomeration. The ingredients of food powders are often heat sensitive and additionally food applications require excellent instant properties.

The above technology is therefore frequently applied for food agglomeration. For instance most cocoa beverages are agglomerated by steam-jet agglomeration [32].

Steam-jet agglomeration is performed by exposing particles to a jet of saturated steam. The condensing steam provides the necessary binder liquid to increase the adhesion forces between colliding particles [65]. Approximately 25–50% of the water contained in the steam condenses in the agglomeration zone while the powder moisture content increases from 0.1–2% to 3–5% [53].

Particles made of amorphous water-soluble polymers can be easily agglomerated by steam agglomeration because of the water–amorphous substance interaction and the high viscosity of the generated bridges between the particles. The water condensing on the particle surface migrates into the amorphous matrix and the particle surface temperature increases up to 80–90 °C. In consequence the viscosity of the amorphous substances at the particle surface decreases below 10^8 Pa s whereas the particle inside remains dry. Accordingly degradation reactions in the particle inside are reduced. During impact the surface of colliding particles is deformed and a rapid sintering leads to a stable highly viscous bridge between the collision partners. Starting from amorphous particles, the initial particle size can be quite large because of the high stability of the bridges formed between the particles.

Crystals can only be agglomerated if they are milled to smaller particle diameters prior to agglomeration [53]. In addition to condensation of steam on the particle surface, droplets are also formed by condensation of vapour in the gas phase. The crystalline substances dissolve in the condensing steam. In case of agglomeration of mineral salts the dissolved ions do not lead to a significant increase in viscosity. Accordingly true liquid bridges having a low stability are formed between colliding particles. The crystalline substance dissolves in the condensed water and forms thin but stable crystalline bridges upon drying.

Since the difference in velocity between single particles is below 0.2 m/s [53] and the particle concentration in the agglomeration zone is low, the collision frequency is also low. Thus, the presence of loose agglomerates in the milled and chilled powder prior to its exposure to steam is desired. In such brittle agglomerates particles are already in close contact before they enter the agglomeration zone where they are exposed to the condensing steam. Milling of the powder prior to agglomeration supports the formation of such loose pre-agglomerates.

Steam jet agglomeration is only applicable to water-soluble powders. It delivers brittle agglomerates with excellent instant properties. Thus it is mainly used for manufacturing all types of instant food powders that have to dissolve rapidly and that might contain heat sensitive substances.

3.1.3. Fluid-bed agglomeration of food, pharmaceutical, detergent powders and fine chemicals

Fluid-bed agglomeration is widely used by the chemical, food and pharmaceutical industry to improve the instant properties and the flowability of various powder products. Food powders such as dairy powders, culinary powders for vending machines, fine chemicals such as vitamin mixes, detergent powders and pharmaceutical excipients are agglomerated by fluid-bed agglomeration. Fluid-bed agglomeration of food powders aims at increasing the flowability (especially in vending machine applications) and improving the instant properties and colour (in retail products) of the powder. Pharmaceutical powder blends are agglomerated in fluid-beds prior to tableting in order to improve dosing accuracy during tableting and to avoid demixing during transport processes. However, despite the diversity of applications similar process conditions are applied for products characterized by the same polarity and supra-molecular structure. In the frame of fluid-bed agglomeration the powder is fluidized by an upward-directed air stream and 2–20% of a binder solution is sprayed on the

moving particles. For amorphous polar (water-soluble) powders it is mostly sufficient to atomize pure water on the fluidized particles. Crystalline water-soluble particles are frequently agglomerated by spraying a viscous aqueous lactose, mannitol or hydrocolloid solution on the fluid-bed. Hydroxypropyl cellulose, polyvinylpyrrolidone and starch/lactose solutions are viscous polymeric binders that are frequently used for fluid-bed agglomeration of pharmaceutical powder blends containing a high concentration of crystalline active substances. Furthermore all type of powders can be agglomerated by spraying on their solidifying binder melts such as triglyceride mixes and polyethylene glycol (PEG). The generated liquid bridges are then solidified by chilling.

The agglomeration of detergent powders using the reactive binder linear alkybenzene sulphonic acid (LAS) is a specific case. The liquid binder solution contains a major amount of LAS which is neutralized through a reaction with solid caustic soda or sodium carbonate. The alkali substance is often a component of the powder blend [18]. During the neutralization reaction gas and water are liberated and the viscosity of the binder increases significantly. The viscosity of the amorphous bridges established between the particles of the base powder depends on the degree of neutralization and the water content. Typically 50–70% LAS have to be neutralized for adhesion of the particles.

Alternatively solutions of alkyl sulphonate, alkyl ether sulphates, olefin sulfonates and methyl ester sulphonates are also used as binder during detergent granulation [8]. These liquid binders are sprayed on a powder mix containing for example zeolites and salts such as sodium silicate, sodium citrate and triphosphates. During agglomeration of detergents maximization of the liquid content (and thus of the detergent content) of the granules is intended. The majority of these binders form amorphous viscous bridges between the particles which are then transformed into glassy solids by drying.

Independently on the base powder and the used binder liquid, the injected binder droplets colliding on the particle surface allow the development of liquid or sinter bridges between colliding particles.

Sometimes melted lipids are used as binders [52]. Melted lipid bridges (e.g. mono-, di- or tri-glycerides) are stabilized by lipid crystallization upon cooling using cold fluidising air. The lipids have to be cooled below their melting temperature in order to prevent caking of the agglomerated powder during the following storage.

For agglomeration of water-soluble amorphous substances or powder blends containing such substances mainly pure water or water-based low-viscous solutions are used as liquid binder. When water-based binder solutions are used, the fluidising air typically has a temperature comprise between 70 and 100 °C and acts as heat and moisture carrier. The moisture evaporating on the particle surface cools the powder down and the powder temperature approaches the wet-bulb temperature. The powder temperature thus drops from 40–50 °C to 20–40 °C during the injection of the aqueous binder. During binder injection the adhesion forces between particles might increase steadily due to the increasing humidity in the air in the fluid-bed. Thus, it is a common practice to increase the air velocity during the liquid injection to maintain the fluidisation state of the powder. After the injection of an aqueous binder the agglomerates are dried to a moisture content of 1–5% and further cooled below their glass transition temperature to avoid caking during storage.

Water droplets colliding on agglomerates containing water-soluble substances penetrate into the porous particle structure in less than one tenth of a second [14]. Although the droplet might already have been penetrated into the pores, some liquid will remain on the particle surface. Generally droplets which impinge on non-porous or saturated particle structures remain longer on the surface. Polar amorphous substances dissolve in these water residuals while simultaneously water migrates into the amorphous matrix. Due to the absorbed water the glass transition temperature of the amorphous substance strongly decreases and the amorphous matrix undergoes glass transition. In the

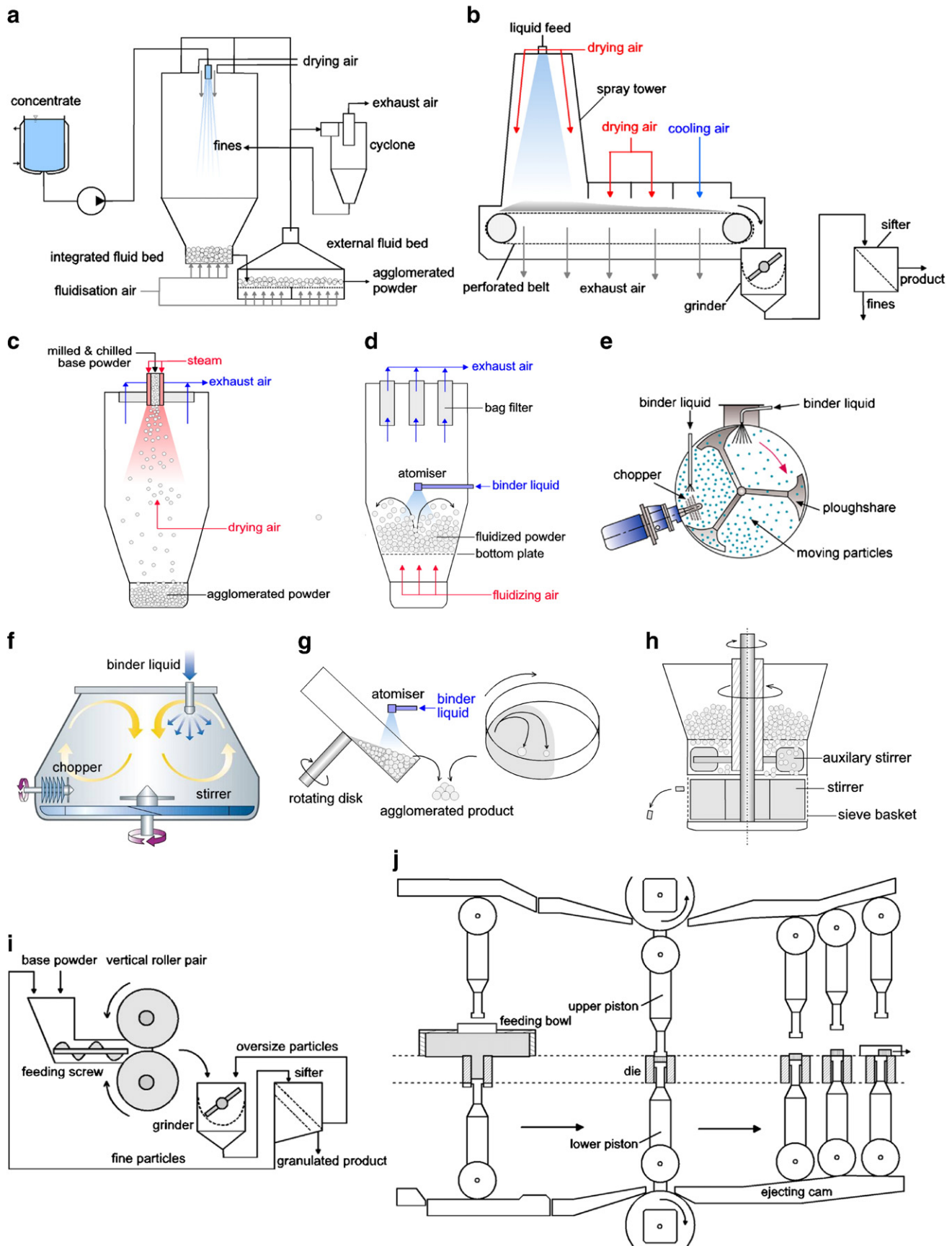


Fig. 7. Overview on most common agglomeration processes applied by the food, pharmaceutical, chemical and detergent industries. a) Multi-stage spray drying w. recirculation of fines. b) Integrated belt drying ("Filtermat drying"). c) Steam-jet agglomeration. d) Fluid bed top-spray agglomeration. e) Mixer agglomeration in high shear mixer (courtesy Lödige GmbH Paderborn/G). f) Wet granulation in vertical granulator (courtesy Glatt GmbH Binzen/G). g) Disk agglomeration. h) Extrusion of wet powder masses (according to Bepex GmbH Leingarten/G). i) Roller compaction. j) Tabletting in double-punch rotary tablet press.

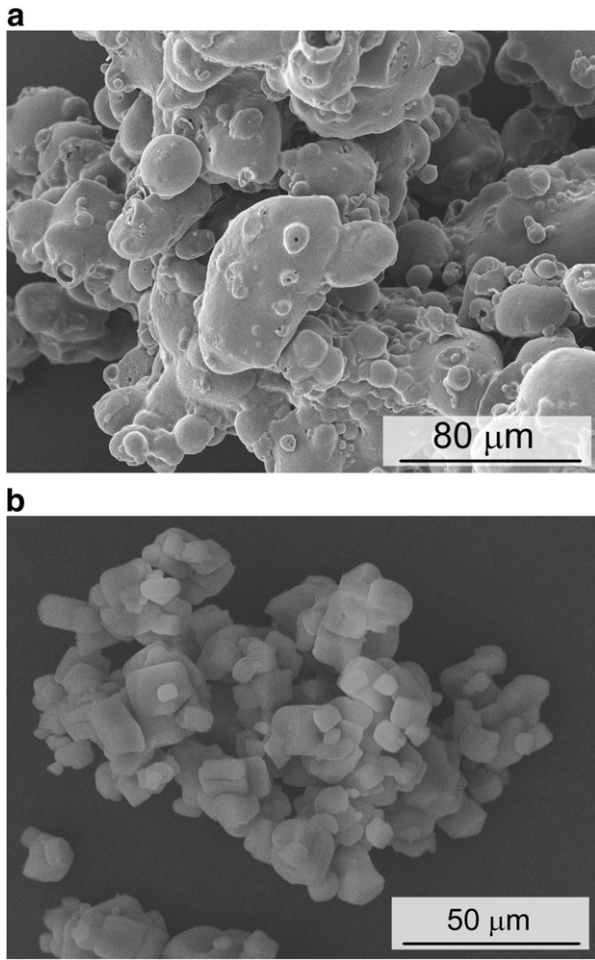


Fig. 8. Scanning electron microscopic images of amorphous milk powder and sodium chloride which were both agglomerated during spray drying. a) Spray dried skim milk powder. b) Spray dried crystalline sodium chloride.

mean time the viscosity of the residual liquid increases due to the dissolved amorphous substance.

In the initial phase of the wetting process, the concentration of polar amorphous solids present in the water remaining on the particle surface is low. Thus, the liquid viscosity is low and true liquid bridges stabilized by capillary forces are formed between colliding particles. With increasing solid content in the droplet, the viscosity increases and adhesion becomes a time-dependent process. At medium or high viscosities adhesion is mainly based on the development of sinter bridges between the particles. Dry particles impacting on the wetted particle surfaces can sinter together with the plasticised material present at the particle surface. In addition, such plasticised regions are easily deformed and, thus, increasing Van der Waals forces between colliding particles also play a role for adhesion. The amorphous substance in these bridges might crystallize during agglomeration, post-drying and storage. In this context, Tardos et al. [58] investigated the structure and crystallinity of bridges build between pharmaceutical particles.

The adhesion mechanism is different when agglomerating water-soluble crystalline powders in a fluid-bed by atomizing water on the moving particles. The crystalline substance partially dissolves in the droplets impacting on the crystals surface and almost no water migrates into the crystalline molecular matrix. Thus, a concentrated solution remains on the particle surface. If another particle collides with the wetted particle, a liquid bridge might be formed between the two particles. In the case of low molecular substances this liquid

bridge often has a low viscosity and the obtained agglomerates are small and fragile. On the other hand, low viscous liquid binder (e.g. pure water) can be easily distributed within the fluidized particle bed due to the friction between colliding particles and the fact that the main part of the liquid remains on the particle surface. During drying of wet agglomerates the liquid bridge is transformed into a stable solid bridge by evaporation of water and re-crystallization of the dissolved substances.

Alternatively to spraying, a melting powdered binder can be added to the particles prior to fluid-bed agglomeration. By fluidizing the powder with hot air the binder is melted or plasticised and the particles adhere to each other forming rather spherical agglomerates. An example for such agglomeration process is the agglomeration of pharmaceutical powder blends with polyethylene glycol (PEG) or waxes.

Fig. 9 shows Scanning Electron Microscopy (SEM) images of different powders agglomerated in a batch-wise operated fluid-bed.

Depending on the process conditions the produced agglomerates are more or less spherical and have a comparable high stability. Increasing fluidization velocity and bed height increases the friction within the fluidized powder bed. Higher friction in the bed, lower viscosity of the liquid binder and larger diameter of the primary particles generally delivers more spherical agglomerates (see Fig. 9b and c). In the case of smaller particles, amorphous water-soluble polymers which significantly increase the bridge viscosity and a low shear-stress in the bed irregular shaped agglomerates (see Fig. 9a and d) are obtained.

3.1.4. Mixer, drum, pan or disk agglomeration of food, pharmaceutical, detergent powders and fine chemicals

During mixer agglomeration the powder is fluidized by fast-rotating stirrers or paddles. In the frame of pan, disk or drum agglomeration the particles are kept in movement by the rotation of the container. During disk agglomeration the powder is moving on an inclined rotating disk (resembling a cataract). Up to 15% of a liquid binder is sprayed on the surface of the moving particles. Alternatively the bulk liquid binder is poured on moving particles and distributed due to the shear stress in the moving powder bed. Another possibility is to add powdered binder to the base powder and to melt the binder by heating the moving particle mix.

Mixer, pan or drum agglomerators are very suitable for agglomeration of fine crystalline water-soluble powders. Agglomeration of water-soluble particle with low glass transition temperature is more difficult due to the strong adhesion forces between moist amorphous particles which lead to a rather broad particle size distribution and the formation of a crust on the equipment surface.

Mixer agglomeration is frequently applied for agglomeration of detergent formulations. The detergent base powder is agglomerated using LAS as binder. During 0.5–3 min, 5–15% of liquid binder is sprayed on the mechanically fluidized particles. Often detergent powders are agglomerated in two successive continuously operated mixers. In the first mixer the main liquid amount is added and distributed by fast rotating mixing tools. In the second mixer the agglomeration is performed at lower rotational speed of the container or the mixing tools. These agglomerated powders are either sold as free flowing powders or are compressed into detergent tablets having 1, 2 or 3 layers and sometimes smaller round tablets are glued on these larger tablets.

Pharmaceutical powders foreseen for tablet manufacturing are agglomerated in mixers to improve compactibility [63] and dosing accuracy during tableting. In the pharmaceutical industry this process is referred to as wet-granulation. Kristensen and Schaefer (1987) [30] give a good overview concerning wet granulation of pharmaceutical powders. The pharmaceutical powder blends are commonly composed of the active component (e.g. Ibuprofen) and a powdered agglomeration aid or excipient. Microcrystalline cellulose (MCC), which is water insoluble and partially crystalline, is frequently used as pharmaceutical excipient. It

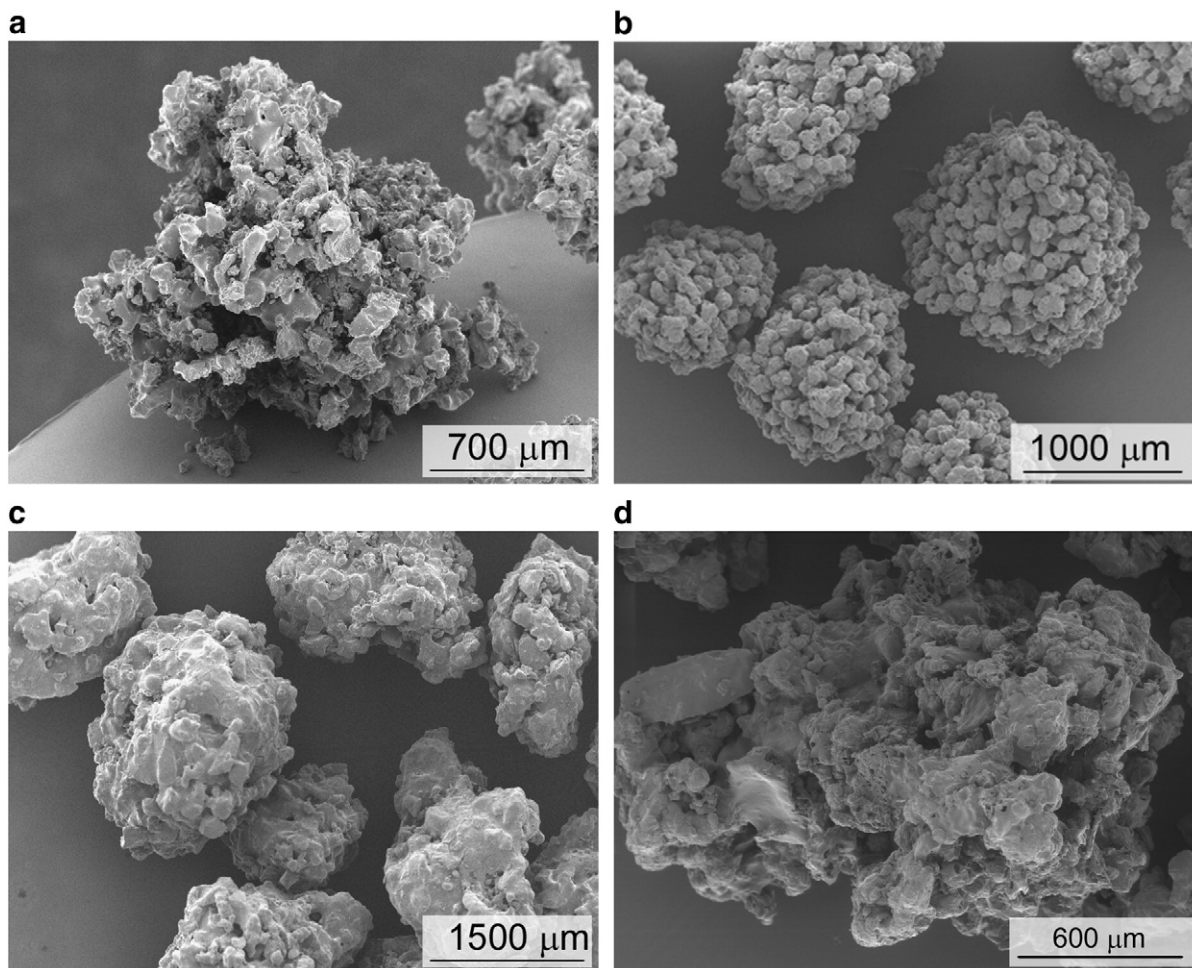


Fig. 9. Agglomerates produced in a batch-wise operated fluid bed (SEM pictures: a) Dextrose syrup agglomerated with water. b) Mannitol agglomerated with PEG6000: [4]) c) Instant beverage agglomerated with water. d) Food powder agglomerated with water).

absorbs water added as binder and thus forms a plastic mass sticking the remaining particles together. Besides pure water, other solutions can be used as binder for agglomeration of pharmaceutical powders. For instance polyvinylpyrrolidone (PVP), sodium carboxymethyl cellulose and hydroxypropyl cellulose (HPC) solutions are used as binder by the pharmaceutical industry. Mixer agglomeration of pharmaceutical powder mixes is typically performed in batch mixers equipped with a vertical shaft with two or three horizontal blades (see Fig. 7f).

Mixer agglomeration of pharmaceutical powders is sometimes also performed as melt agglomeration. Polyethylene is used as melting binder which becomes liquid when the powder mass is heated. Alternatively a PEG melt can also be sprayed on the moving particles. After chilling the PEG binder solidifies.

The largest product volumes agglomerated by pan agglomeration are iron ores. 2% bentonite is added to the humid iron ore dust ($x_{50,3} < 50 \mu\text{m}$). While the particles are agglomerated in rotating inclined pans, additional water is added. The wet agglomerates are dried and sintered.

Also large fertilizer quantities are agglomerated in rotating containers. During agglomeration of fertilizers an ammonium nitrate melt is sprayed on calcium–magnesium carbonate (called dolomite). Fertilizers are sometimes also agglomerated in inclined pans. Water or an aqueous fertilizer solution is sprayed on a cataract of moving particles.

Mixer agglomeration is also applied to some food powders such as culinary powders, powder blends for preparation of desserts, protein powders, starch, bakery mixes and flavour powders. For example,

water, water-based binder solution containing carbohydrates or hydrocolloids, lecithin, molasses or melted fat can be used as binders.

Agglomerates produced in mixers typically have a diameter between 0.5 and 5 mm. During agglomeration of powders in high shear mixers the comparable high shear-stress generates dense and relatively spherical granules (see Fig. 10a).

Pan or disk agglomeration, which is comparable to mixer agglomeration, is applied to iron ores prior to extracting the metal and to fertilizers. Because pan or disk agglomerators are commonly operated as open systems, hygiene concerns are raised and they are therefore rarely used in the food and pharmaceutical industries.

Granules produced by disk or pan agglomeration typically have a diameter between 2 and 10 mm and a spherical shape because of their rotation on the turning disk. The density of mixer or pan agglomerated granules strongly depends on the width of the particle size distribution of the initial powder and the stability of the primary particles. The density of the surface layer of agglomerates manufactured in high shear mixers and pan agglomerators is on average higher than that of agglomerates produced in fluid-beds.

For agglomeration of large product quantities in mining, detergent and fine chemicals industries, continuous mixers or pan agglomerators are installed. Continuous agglomeration under addition of moisture or binder melts is followed by drying of the obtained agglomerates using continuously operated fluid-bed driers. In the food and pharmaceutical industry batch mixers are mainly used because of smaller batch sizes, better traceability and a lower risk of cross

contaminations. The produced agglomerates are dried in batch-wise working ovens or fluid-bed driers.

3.2. Pressure agglomeration of food powders

In the absence of binder liquid or when there is insufficient plasticisation of the substance building the particles, the adhesion forces between colliding particles are very weak. However, by increasing the force pressing the particles together, it is possible to agglomerate powders without addition of binder and significant plasticisation of the particles. The stress exerted on a confined powder volume deforms the particles and thus leads to increasing Van der Waals forces. Depending on the nature of the material this might be followed by a successive sintering at the contact points between the particles. Such agglomeration processes in which the particles are subject to a significant pressure are referred to as pressure agglomeration. Extrusion, tableting and roller compaction are typical pressure agglomeration processes.

3.2.1. Extrusion of wet powder masses

Extrusion of wet powder masses is known as “wet massing” or “sieve extrusion”. Extrusion of wet powder masses is applied to food additives and ingredients like aspartame, sorbitol, pectin, xanthan gum, gelatine, icing sugar, gluten, yeast extracts, soy bean powders, corn syrups and whey powders. Food powders like instant tea, sweet beverages, seasonings, and culinary binders are agglomerated by extrusion of wet powder masses. Furthermore also pharmaceutical

powders foreseen for tableting are extruded to improve their flowability which is required for exact dosing [40]. In addition pharmaceutical powders are extruded and following spheronized. The obtained spherical pellets are filled into capsules.

Large quantities of animal-feed products are also extruded into cylindrical pellets.

Last but not least wood and waste particles are also pressed through perforated flat dies. Die obtained cylindrical pellets are incinerated.

Before extrusion the powder mix can be wetted with 3–20% water or melted fat in a kneader or powder mixer in order to increase the adhesion forces between the particles. The particle mass is pressed by a piston or agitator through a die consisting of various holes. This is performed in flat-die peletting presses, extruders with circular die plates (see Fig. 7h) and horizontal single or double screw extruders. The friction between the wet powder mass and the wall of the die holes generates the pressure needed for forming stable agglomerates. The obtained product strand is then separated into individual agglomerates by cutting them into small cylinders or it is broken into single agglomerates by forces like gravity or inertia.

When extruding wet powder masses containing a high amount of amorphous materials, it is difficult to avoid blockages of the orifice and to control the agglomerate length. During the process, the extruder might get warm because of the friction between the rotating tools, the powder mass and the surface of the die. Thus, the viscosity of amorphous substances changes and the adhesion between the material and the rotating tools or the housing of the extruder strongly increases. Hence, often a sticky crust is formed. Furthermore, the adhesion between single particles increases due to increasing bridge diameter between them. Forces like gravity or inertia cannot easily break the strands and consequently longer agglomerates are obtained. Cutting tools are sometimes installed at the orifice to separate single granules from each other.

In comparison, fine water-soluble crystalline powders can be easily agglomerated by extrusion. The powder is milled and wetted with water in mixers. The humid powder mass is extruded and the obtained cylindrical granules are dried in fluid-beds or ovens.

Cylindrical agglomerates with a dense cylinder surface are obtained by extrusion of wet powder masses.

3.2.2. Roller compaction

Roller compaction is primarily used for the agglomeration of chemical and pharmaceutical powders. However, also some food ingredients such as crystalline sucrose, sodium chloride, monosodium glutamate and fibres are agglomerated by roller compaction to reduce their transport volume and to improve their flowability and optical appearance. Roller compaction is also applied as low-cost encapsulation process to flavour powders.

In roller compaction a high pressure is exerted on a continuous powder stream which moves through the gap between two counter-rotating rollers. The base powder is fed into the gap between the two synchronized counter-rotating rollers using a force feeder (or sometimes just gravity). While passing the gap between the two rollers the volume available decreases and the powder is compressed into briquettes or a continuous ribbon. The wall friction between the powder and the roller surface, which increases with increasing pressure, draws the particles into the gap between the two rollers. With increasing pressure particles deform and adhere to each other due to increasing Van der Waals forces. When a flat roller surface is used, a thin, flat ribbon sheet is obtained whereas a roller surface with large cavities generates briquettes. The two rollers turn at a speed of up to 20 rpm. The compacting pressure is adjusted either by increasing the feeding rate or by adjusting the distance between the two rollers. According to Johanson [26], it is possible to increase the pressure generated within the roller gap by using rollers with rough surfaces which increase the throughput of the system.

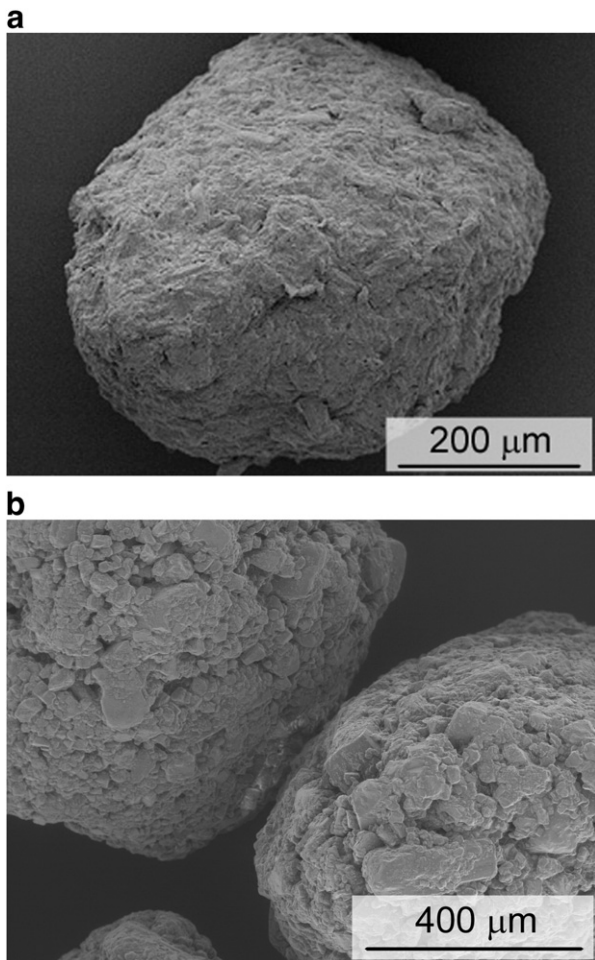


Fig. 10. Agglomerates produced in a high shear mixer and a pan agglomerator. a) Micro crystalline cellulose + PVP (3% PVP solution, high shear mixer) [Benali et al. [5]]. b) Food powder agglomerated in a pan.

After the compaction process the obtained endless ribbons or briquettes are grinded to a final particle size of 0.2 to 3 mm in a so-called “granulator” — a sieve basket in which a stirrer oscillates or rotates with 200–300 rpm. Alternatively, toothed crushers can be used for crystalline materials to break the obtained ribbons. To optimise the particle size distribution, a two-step grinding process can be performed. The amount of fines generated might vary between 5 and 50%. The fines produced during grinding are added back into the feeding hopper of the compactor.

Agglomerates manufactured by roller compaction have a particle diameter between 0.5 and 3 mm. The resulting granules are sharp-edged and irregular shaped. Compared to porous agglomerates obtained by fluid-bed agglomeration, their dissolution rate is significantly lower due to their high density.

3.2.3. Tableting of pharmaceutical, detergent and food powders

Tableting is a pressure agglomeration process which transforms a free flowing powder into a defined shape. Tablets are mechanically stable, and compared to other agglomerates, often are characterized by a low dissolution rate due to their high density and large dimensions.

Tableting is applied by the pharmaceutical, food and detergent industries for agglomeration of various types of powder.

The pharmaceutical industry produces tablets which are meant to be swallowed and which should disintegrate when reaching the human gastro-intestinal track. Effervescent tablets are also available to the consumer. Commonly they are consumed after dissolution in water. Prior to tableting the active substance is blended with different excipients such as amorphous pre-gelatinized starch, microcrystalline cellulose, inulin, spray-dried semi-crystalline lactose (e.g. 85% crystalline), crystalline α -lactose monohydrate and crystalline dicalcium-phosphate. In some cases (e.g. acetylsalicylic acid tablets or paracetamol tablets) the active component accounts for the largest amount of the powder mass. Pharmaceutical tablets provide an exact pre-dosed powder portion. By selecting an appropriate density or coating of the tablets their disintegration can be modulated and a controlled release of the active substance can be achieved. Pharmaceutical tablets are relatively small compared to other types of tablet, and can have pillow-like, ellipsoidal, cylindrical or spherical shapes to ease swallowing.

Detergent tablets are made of a base powder which is agglomerated in a mixer prior to tableting. The base powder is composed of zeolite, salts and minor components. These are agglomerated with a liquid phase containing anionic and non-ionic surfactants. The solidified liquid binder builds an amorphous phase in which the solid base powder particles are embedded. The dried granules are compressed into a tablet. Detergent tablets mostly have the shape of squares, and are rather large (2–3 cm).

Food tablets which are dissolved prior to consumption represent a pre-dose product quantity in a specific and easily recognisable shape. Sweets or tableted performance products for sports men are made of dextrose, mannitol, trehalose and other carbohydrates or sugar alcohols. Due to their high density they slowly dissolve in the mouth. Typical food tablets have a diameter between 3 and 40 mm. Confectionary tablets are offered in a large variety of different shapes and sizes: hearts, hollow cylinders, pillows, flowers and confectionery tablets in the shape of clocks are even available. In seasoning cubes and squared bouillon tablets fat is often used as binder substance. Contrarily to pharmaceutical powders, food powders are not agglomerated prior to tableting due to cost reasons.

Effervescent tablets contain amorphous or crystalline citric or ascorbic acid and calcium bicarbonate. During dissolution the caustic and acid components react with each other forming salt and carbon dioxide.

Amongst the largest and most dense tablets produced are sodium chloride tablets for water treatment in large plants.

Tableting is also applied by the automotive and ceramic industries to transform a free flowing powder into a predefined shape which is then hardened by sintering. The produced pieces are used in manufacturing several type of equipment. The automotive industry compresses distloy, tungsten carbide, steel and copper powders to different shapes. In the ceramic industry mainly aluminium oxide (alumina), zirconia and steatite are tableted before sintering.

Finally also radioactive materials (e.g. plutonium) are tableted for application in nuclear power plants.

Despite these different applications of tableting the technology remains for all industries more or less the same. Depending on the crystallinity, the plasticiser content and the corresponding mechanical material properties, different compression parameters have to be applied. Amorphous materials containing a significant plasticiser content and substances deforming plastically at a low or medium yield strength are comparable easy to compress.

Prior to tableting, the different powdered components are mixed in a powder mixer. In the pharmaceutical industry this powder mix is most frequently first agglomerated in a fluid-bed or in a high-shear mixer in order to improve the dosing accuracy during the following tableting process. In the pharmaceutical industry, often batch-wise operated vertical granulators (see Fig. 7) are used for this agglomeration step. The agglomerated moist powder used for tableting is dried to avoid caking, chemical and enzymatic reactions and microbiological spoilage during the intermediate storage. Such a pre-agglomerated powder can be more easily compacted into tablets and has an improved flowability compared to the initial powder mix. However,

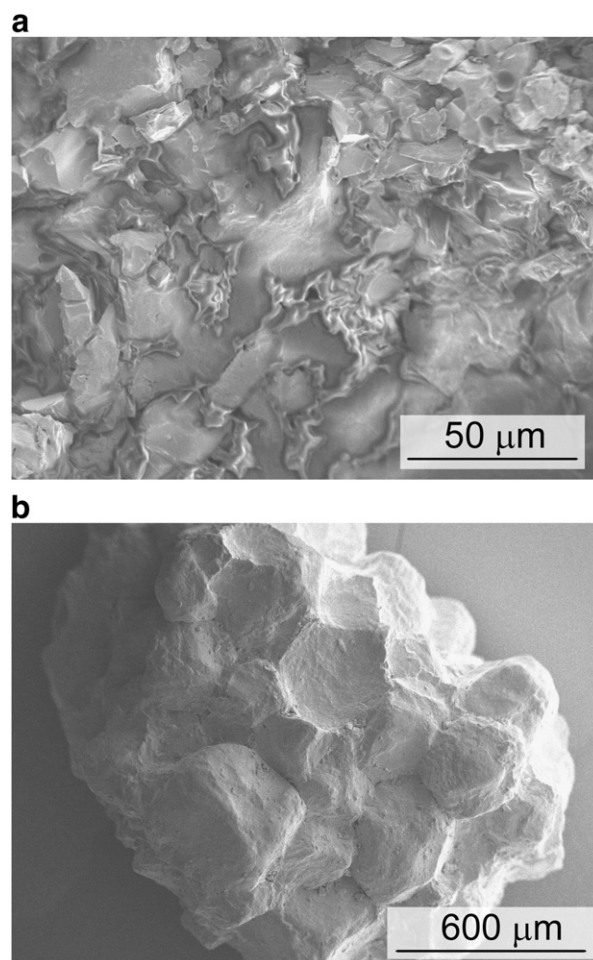


Fig. 11. Agglomerates produced in a roller compactor. a) Compacted dextrose syrup (DE21). b) Compacted sodium chloride (provided by M. Hartmann/Nestec).

some powders can sometimes be tableted directly without any preceding additional agglomeration step, so that manufacturing costs are reduced. Alternatively only a part of the final formulation is granulated, dried and later blended with the remaining ingredients prior to tableting [59]. Such an approach is especially useful to prevent the degradation of heat sensitive ingredients like flavours, encapsulated essential oils, vitamins or heat sensitive drugs during the necessary drying.

During the tableting process the powder is filled into dies and compacted to tablets with a defined shape. The design of the feeding tools has to be adapted to the powders flowability which depends on the diameter, the shape and the surface viscosity of the particles. After filling the die, the powder is compressed between two vertically moving punches (see Fig. 7j). For fat-containing food powders the powder is pressed by a piston against a rotating horizontal counter plate. Compression is often a two-step process: a pre-compression precedes the main compression in order to facilitate the release of air entrapped between the particles and to increase the compression time for viscoelastic particles. The surface of the compression tools has to be adapted to the stickiness and mechanical properties of the material. Particles containing a significant amount of amorphous materials with a viscosity below 10^8 Pa·s tend to adhere to the surface of the pistons. The same is observed for powders containing a larger amount of crystallized lipids.

For tableting, the mechanical properties of the particles are determining the pressure generated during the densification of the powder and the achieved deformation. Amorphous materials gener-

ally behave viscoelastically. In this context it has to be considered that the mechanical properties of water-soluble amorphous substances are a function of temperature and moisture content of the powder. At high compression speed and if the material is significantly below its glass transition temperature amorphous substances fracture leading to a smaller particles. At low compression speed and close to their glass transition temperature they exhibit more viscous properties and deform plastically. The viscoelastic compression increases the Van der Waals forces between particles. The final tablet hardness depends on the compression force, the time during which the pressure is applied (dwelling time) and the viscoelastic material properties. Rees and Rue [49] thus reported that the achieved deformation of lactose, starch and cellulose powders is time dependent. All these substances are behaving viscoelastically [38]. The viscoelastic properties of amorphous water-soluble substances such as amorphous lactose, dextrose syrup, maltodextrine, amorphous sugar alcohols (e.g. amorphous mannitol), pre-gelatinised starch or amorphous organic acids (e.g. citric acid) are a function of temperature and moisture content. Increasing the temperature and/or the moisture content decreases the elastic modulus and the viscosity of the amorphous matrix. The hardness of tablets made of such substances depend on the moisture content of the powder, the temperature during compression and the pressure/time profile applied during compression [37,42,43]. These considerations are supported by published experimental data obtained in the frame of various tableting trials: Esezobo and Pilpel [15] found that the tensile strength of tablets increases with increasing temperature. According to Shukla and Price [56] the

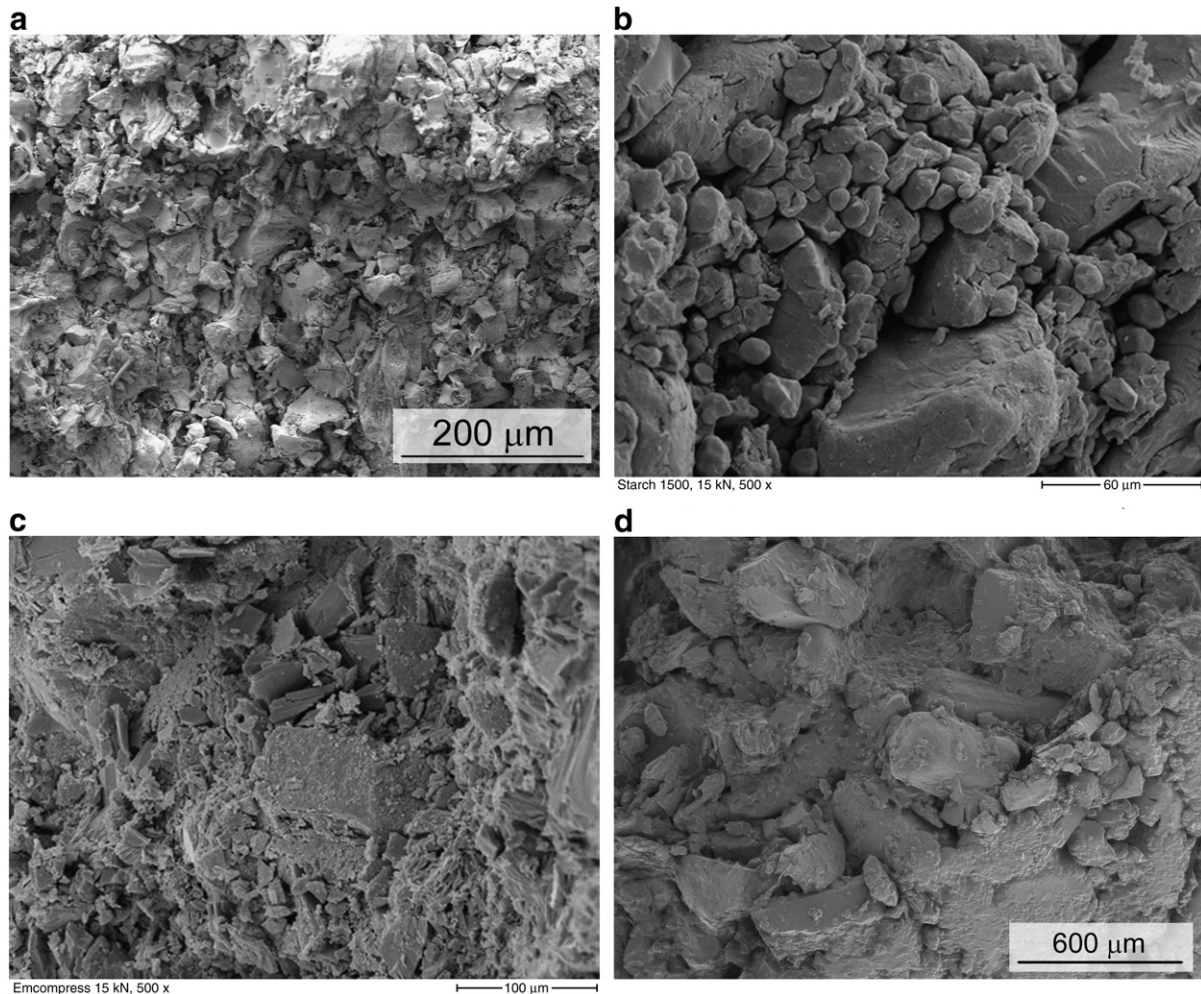


Fig. 12. Electron microscopic images of tablets made of different food and pharmaceutical excipients. a) Dextrose syrup tablet. b) Pharmaceutical tablet made of physically modified starch (Cunze, 2003 [10]). c) Pharmaceutical tablet made of α -lactose monohydrate (Cunze, 2003 [10]). d) Food tablet made of sodium chloride and other ingredients.

pressure required for tableting direct compressible dextrose-based powders decreases with increasing moisture content of the powder. Picker investigated the tableting of different amorphous powders [45]. The crushing force of the produced tablets was high if the glass transition temperature was reversibly exceeded during compression.

A number of tablets are made of crystalline water-soluble powders like crystalline sucrose, mannitol, lactose or sodium chloride. Crystalline materials, e.g. mannitol, fracture and deform plastically [27]. Provided that the air within the powder bulk can be released effectively during compression, the hardness of tablets composed of crystalline lactose, mannitol, sodium chloride, and metals depends only on the maximal pressure applied during compression. Crystalline materials do not absorb large water quantities and their mechanical properties are supposed to change only very little with increasing temperature. Thus, relative humidity of the surrounding air and variations in temperature should not affect the compression significantly. Nevertheless, it seems that moisture plays a role for tableting of some crystalline water-soluble materials. For sodium chloride tablets Rees and Hersey [48] found that the tablet stability decreases with increasing moisture content of the crystalline raw material. Ahlneck and Alderborn [2] investigated the effect of air humidity on the volume reduction properties and the tablet strength for some crystalline materials like sodium chloride and sucrose. The tensile strength of the obtained tablets was, depending on the investigated material, either in- or decreasing with increasing moisture content of the air prior to tableting. While the effect of moisture on the tensile strength of sodium chloride tablets was small, a significant increase in tensile strength was observed for crystalline lactose, sodium citrate and crystalline sucrose. The observed effect of moisture on the tensile strength of tablets made of water-soluble crystalline materials might be explained by a partial dissolution of the crystals while exceeding the critical relative humidity or by capillary condensation during compression. Furthermore, a partial transformation of the crystalline material into its amorphous state due to high internal friction during densification can not be excluded.

Microcrystalline cellulose is a porous, water-insoluble, hygroscopic binder which is frequently used in the pharma-industry. It has a semi-crystalline supra-molecular structure. Exposed to high relative humidity microcrystalline cellulose absorbs significant quantities of water and swells. During compression it deforms plastically [57]. At higher water content a significantly lower compression force is required to obtain certain tablet hardness.

In powders which contain a significant amount of lipophilic binder (e.g. solid fat, waxes or stearate), the plastic deformation of the binder, leading to increasing Van der Waals forces, determines the tablet hardness. For such formulations, the obtained tablet hardness mainly depends on the mechanical properties of the binder, which are a function of temperature and tableting pressure. Fig. 12 shows electron microscopy images of different tablets. All structures are comparably dense. Obviously the glassy dextrose syrup particles (Fig. 12a; compressed 30–50 °C below T_g) the lactose crystals (Fig. 12c) fractured during compression. The visco-elastic amorphous particles build of physically modified starch (Fig. 12b) are significantly deformed. In the tablet structure shown in Fig. 12d vegetable fat acts as water insoluble plastically deformable binder sticking the particles together.

4. Conclusions

Categorizing the substances building the base powder into on one hand amorphous and crystalline materials and on the other hand polar and apolar materials helps to identify similarities between agglomeration in different industries. Furthermore one can distinguish between agglomeration processes during which the particles collide randomly and adhere to each other if their surface viscosity is below 10^6 Pa s, processes during which static sticky particles sinter together during drying and pressure agglomeration processes leading

to increasing van der Waals forces between the particles. Accordingly the processes and the various base powders used in different industries can be compared with each other.

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PatBase - Search results 27 April 2015

Search query (9): [SP]: 1. a process for preparing a bouillon tablet having two or more layers comprising the steps of: forming a first layer of the tablet comprising a mixture of two or more of salt, taste enhancer, sugar, and fat (or oil) forming a second layer of the tablet by adding to the first layer a mixture comprising one or more of herbs, garnishes, vegetable pieces, and spices and compressing the two layers to form the table, wherein the tablet is formed using a rotary double-punch press. 2. a process according to claim 1, wherein the one or more of herbs, garnishes, vegetable pieces, and spices are coated with fat or oil. 3. a process according to claim 2, wherein the fat is hydrogenated palm oil. 4. a process according to claim 1, wherein the compression force applied to form the first layer is less than 40 percent of the compression force applied to form the tablet. 5. a process according to claim 1, wherein the compression force applied to form the first layer is 0.1 to 1.5 kn. 6. a process according to claim 1, wherein the compression force applied to form the tablet is 0.8 to 6 kn. 7. a process according to claim 1, wherein the second layer is 1-35 percent by weight of the tablet. 8. a process according to claim 1, wherein the mixture comprising one or more of herbs, garnishes, vegetable pieces, and spices has a moisture content below 7 percent by weight. 9. a process according to claim 1, wherein the first layer is formed by adding the mixture of two or more of salt, taste enhancer, sugar, and fat (or oil) into a die-bore of the rotary double-punch press and then applying a compression force. 10. a process according to claim 1 9, wherein the mixture comprising one or more of herbs, garnishes, vegetable pieces, and spices is added to the first layer in the die-bore and a compression force applied. 11. a bouillon tablet of the hard bouillon type prepared by forming a first layer of the tablet comprising a mixture of two or more of salt, taste enhancer, sugar, and fat (or oil), forming a second layer of the tablet by adding to the first layer a mixture comprising one or more of herbs, garnishes, vegetable pieces, and spices, and compressing the two layers to form the tablet, the tablet being formed using a rotary double-punch press. 12. a method for the preparation of a soup, sauce, bouillon, or gravy, or for use as a seasoning comprising adding liquid to a bouillon tablet prepared by forming a first layer of the tablet comprising a mixture of two or more of salt, taste enhancer, sugar, and fat (or oil), forming a second layer of the tablet by adding to the first layer a mixture comprising one or more of herbs, garnishes, vegetable pieces, and spices, and compressing the two layers to form the tablet, the tablet being formed using a rotary double-punch press.

Results: 11

Search history

- Search 1: TAC=(CONSTRUCT~ AND SHELL~ AND GENE~ AND IN~ AND OIL~ AND PALM~) (Results 3)
- Search 2: shell gene oil palm (Results 0)
- Search 3: Shell tickness gene construct in oil plam (Results 0)
- Search 4: oil palm fruit shell tickness (Results 0)
- Search 5: TAC=(OIL~ AND PALM~ AND SHELL~ AND TICKNESS~ AND ALELES~ AND COSTRUCT~) (Results 0)
- Search 6: TAC=(OIL~ AND PALM~ AND FRUIT~ AND SHELL~) (Results 113)
- Search 7: TAC=(bouillon tablet and rotary double-punch press) (Results 0)
- Search 8: TAC=(bouillon tablet or rotary double-punch press) (Results 9)
- Search 9: [SP]: 1. a process for preparing a bouillon tablet having two or more layers comprising the steps of: forming a first layer of the tablet comprising a mixture of two or more of salt, taste enhancer, sugar, and fat (or oil) forming a second layer of the tablet by adding to the first layer a mixture comprising one or more of herbs, garnishes, vegetable pieces, and spices and compressing the two layers to form the table, wherein the tablet is formed using a rotary double-punch press. 2. a process according to claim 1, wherein the one or more of herbs, garnishes, vegetable pieces, and spices are coated with fat or oil. 3. a process according to claim 2, wherein the fat is hydrogenated palm oil. 4. a process according to claim 1, wherein the compression force applied to form the first layer is less than 40 percent of the compression force applied to form the tablet. 5. a process according to claim 1, wherein the compression force applied to form the first layer is 0.1 to 1.5 kn. 6. a process according to claim 1, wherein the compression force applied to form the tablet is 0.8 to 6 kn. 7. a process according to claim 1, wherein the second layer is 1-35 percent by weight of the tablet. 8. a process according to claim 1, wherein the mixture comprising one or more of herbs, garnishes, vegetable pieces, and spices has a moisture content below 7 percent by weight. 9. a process according to claim 1, wherein the first layer is formed by adding the mixture of two or more of salt, taste enhancer, sugar, and fat (or oil) into a die-bore of the rotary double-punch press and then applying a compression force. 10. a process according to claim 1 9, wherein the mixture comprising one or more of herbs, garnishes, vegetable pieces, and spices is added to the first layer in the die-bore and a compression force applied. 11. a bouillon tablet of the hard bouillon type prepared by forming a first layer of the tablet comprising a mixture of two or more of salt, taste enhancer, sugar, and fat (or oil), forming a second layer of the tablet by adding to the first layer a mixture comprising one or more of herbs, garnishes, vegetable pieces, and spices, and compressing the two layers to form the tablet, the tablet being formed using a rotary double-punch press. 12. a method for the preparation of a soup, sauce, bouillon, or gravy, or for use as a seasoning comprising adding liquid to a bouillon tablet prepared by forming a first layer of the tablet comprising a mixture of two or more of salt, taste enhancer, sugar, and fat (or oil), forming a second layer of the tablet by adding to the first layer a mixture comprising one or more of herbs, garnishes, vegetable pieces, and spices, and compressing the two layers to form the tablet, the tablet being formed using a rotary double-punch press. (Results 11)

Title: Process for producing compressed, dehydrated cellular foods

Abstract: Source: US3385715A ABSTRACT OF THE DISCLOSURE A process in which morsels of a freeze-dried cellular food are first rehydrated to a moisture content of about 5% to 13%, compressed together while maintaining the surface moisture of the morsels and the pressure sufficiently high to cause the morsels to adhere during said compression and dehydrated to a moisture content below about 3%, the degree of compression being such that the density of the dehydrated product is in the range of about 0.5 to 0.9 gram per cc.

Classifications:

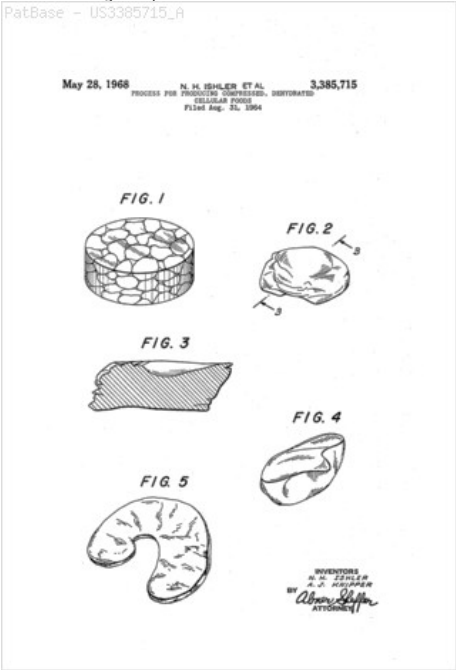
International (IPC 8-9): A23L1/00 A23L1/31 A23P1/10 (Advanced/Invention)

International (IPC 1-7): A23K1/20

CPC: A23L1/0073 A23L1/3103 A23P1/105

European: A23L1/00P10 A23L1/31H A23P1/10B

US: 426/274 426/454 426/506



Family:

Publication number	Publication date	Application number	Application date
US3385715 A	19680528	US19640393819	19640831

Priority:

US19640393819 19640831

Assignee(s): (std): TRONCHEMICS RES INC ; TRONCHEMICS RESEARCH INC

Inventor(s): (std): ISHLER NORMAN H ; KNIPPER ALOYSIUS J

Title: A PROCESS FOR THE PRODUCTION OF DEHYDRATED COMPRESSED FOOD PRODUCTS.

Abstract: Source: EP0326525A1 A process for the production of dehydrated compressed food products, said process comprising the steps, starting from base freeze-dried or dried food products, as individual products or as mixtures of products, of: a) moistening the freeze-dried food product or the freeze-dried mixture of products up to absolute moisture amounts of about 20 percent depending on the kind of food product; b) compressing the moistened product by means of power-consuming machines at pressures between 2 and 100 kg/cm; c) drying the compressed product till obtaining absolute moisture amounts less than or equal to 3 percent . e

Classifications:

International (IPC 8-9): A23B7/02 A23B7/12 A23K1/00 A23L3/44 (Advanced/Invention); A23K1/00 (Core/Invention)

International (IPC 1-7): A23B7/02 A23K1/00 A23L3/38 A23L3/40 A23L3/44

CPC: A23L3/40 A23L3/405 A23L3/42

European: A23L3/40 A23L3/40C A23L3/42

Family:

Publication number	Publication date	Application number	Application date
AT83896 E	19930115	AT19890830019T	19890120
DE0326525 T1	19900412	DE19890830019T	19890120
DE68904081 D1	19930211	DE19896004081	19890120
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EP0326525 A1	19890802	EP19890830019	19890120
EP0326525 B1	19921230	EP19890830019	19890120
ES2012311 T3	19930701	ES19890830019T	19890120
GR3007155 T3	19930730	GR19930400391T	19930225
GR90300067 T1	19910731	GR19910300067T	19910731
IT1219823 A	19900524	IT19880047573	19880127
IT8847573 A0	19880127	IT19880047573	19880127
JP2016960 A2	19900119	JP19890019367	19890127

Priority:

IT19880047573 19880127 EP19890830019 19890120

Assignee(s): (std): AGROFIL COOP SARL ; AGROFIL SOC COOP A R L ; AGROFIL SOC COOP ARL

Assignee(s): AGROFIL SOC COOP A R L CATANIA IT

Inventor(s): (std): LUIGI COSTANZO ; RUIGI KOSUTANZO ; COSTANZO LUIGI

Inventor(s): COSTANZO LUIGI VIA ARTALE ARAGONA 37 I 95126 CATAN ; COSTANZO LUIGI VIA ARTALE ARAGONA 37 I 95126...

Designated states: AT BE CH DE ES FR GB GR LI LU NL SE

Title: Cooking aid of the lump type

Abstract: Source: US6399129B Cooking aid of the lump type having the shape of a lump with a granular surface and comprising a flavoring core and a coating of a binding base and visual components. Also, a process for preparing this cooking aid.

Classifications:

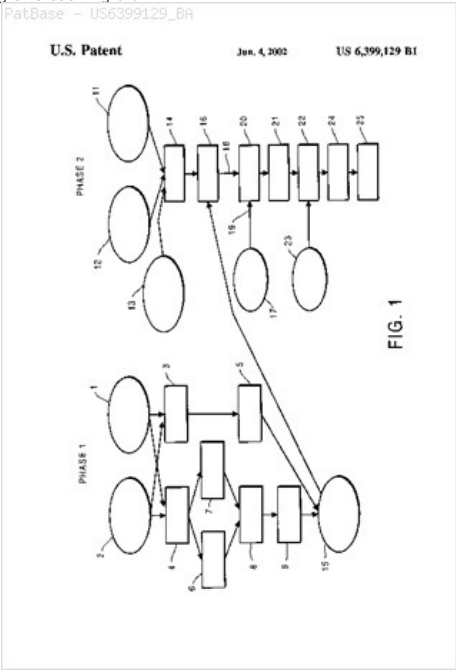
International (IPC 8-9): A23L1/00 A23L1/40 A23L1/48 A23P1/00 A23P1/02 A23P1/08 A23P1/10 (Advanced/Invention); A23L1/48 A23P1/00 A23P1/02 (Advanced/Non-invention); A23L1/00 A23L1/39 A23L1/48 A23P1/00 A23P1/02 A23P1/08 A23P1/10 (Core/Invention)

International (IPC 1-7): A23L A23L1/00 A23L1/03 A23L1/05 A23L1/09 A23L1/22 A23L1/221 A23L1/40 A23L1/48 A23P A23P1/00 A23P1/02 A23P1/08 A23P1/10

CPC: A23L1/40 A23L1/0026 A23P1/027

European: A23L1/00P2D A23L1/40 A23P1/02D

US: 426/293 426/302 426/98 426/99



Family:

Publication number	Publication date	Application number	Application date
AR015396 AA	20010502	AR1998P103250	19980703
AT235170 E	20030415	AT19980201617T	19980515
AU199874062 A1	19990114	AU19980074062	19980702
AU741176 B2	20011122	AU19980074062	19980702
BRPI9802345 A	20000328	BR1998PI02345	19980702
CA2237892 AA	19990104	CA19982237892	19980619
CN1103552 C	20030326	CN19981003223	19980704
CN1205184 A	19990120	CN19981003223	19980704
CZ295363 B6	20050713	CZ19980002122	19980703
CZ9802122 A3	19990113	CZ19980002122	19980703
DE69812482 D1	20030430	DE19986012482	19980515
DE69812482 T2	20030911	DE19986012482T	19980515
EP0888721 A1	19990107	EP19970202060	19970704
EP0888723 A1	19990107	EP19980201617	19980515
EP0888723 B1	20030326	EP19980201617	19980515
ES2193472 T3	20031101	ES19980201617T	19980515
HU9801504 A0	19980828	HU19980001504	19980703
HU9801504 AB	19990329	HU19980001504	19980703
HU9801504 AC	20001228	HU19980001504	19980703
ID20660 A	19990211	ID19980000947	19980702
IL124945 A0	19990126	IL19980124945	19980616
IL124945 A1	20001031	IL19980124945	19980616
JP11069963 A2	19990316	JP19980189195	19980703
MY132944 A	20071031	MY19980003022	19980702
NZ330693 A	20000428	NZ19980330693	19980616
OA10807 A	20030129	OA19980000104	19980703
PL189211 B1	20050729	PL19980327224	19980703
PL327224 A1	19990118	PL19980327224	19980703
PT888723 T	20030630	PT19980201617T	19980515
RU2214140 C2	20031020	RU19980112756	19980703
RU98112756 A	20000327	RU19980112756	19980703
SK91198 A3	19990211	SK19980000911	19980701
TH35354 A	19991015	TH19981002521	19980703
TR9801292 A1	19990118	TR19980001292	19980703
US6399129 BA	20020604	US19980109128	19980702
ZA9805830 A	20000110	ZA19980005830	19980702

Priority:

EP19970202060 19970704 EP19980201617 19980515 CA19982237892 19980619

Assignee(s): (std): NESTLE SA ; NESTEC SA ; SOS ETE DE PRODUITS NESTLE S A

Assignee(s): NESTEC AVENUE NESTLE SA ; Societe des Produits Nestle S A ; NESTEC S A ; SOC PROD NESTLE SA ; SOCIETE DES PRODUITS NESTLE S A VEVEY ; SOS'ETE DE PRODUITS NESTLE S.A. ; DES PRODUITS NESTLE S A SOC ; DES PRODUITS NESTLE SA SOC ; DES PRODUITS NESTLE SOC SA ; GUILLAIN VALERIE ; MAHE YANNICK

Inventor(s): (std): GILLEHN VALERI ; GUILLAIN BEAUVOIS VALERIE ; GUILLAIN V ; GUILLAIN VAL RIE ; GUILLAIN VALERIE ; MAE JANNIK ; MAHE Y ; MAHE YANNICK ; YANNICK MAHE ; VALERIE GUILLAIN ; GUILLAIN VAL EACUTE RIE

Inventor(s): GUILLAIN VAL E RIE ; V GUILLAIN ; VAL RIE GUILLAIN ; VALERIE GUILLAIN BEAUVOIS ; Y MAHE

Designated states: AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU MC NL PT SE

Title: MULTI-INGREDIENT STOCK CUBE FOR PREPARATION OF LIQUID FOODS OR FOOD COMPONENTS

Abstract: Stock cube for culinary preparation of liquid foods or food components in the form of a formed multi-ingredient stock cube, comprising at least two partial volumes which may be visually discriminated and/or discriminated by touch which correspond to food components with differing compositions, in particular those corresponding to meat and vegetable stocks.

Classifications:

International (IPC 8-9): A23L1/00 A23L1/22 A23L1/39 A23L1/40

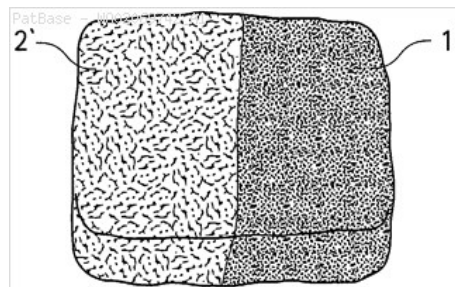
A23P1/02 (Advanced/Invention);

A23L1/00 A23L1/22 A23L1/39 A23P1/02 (Core/Invention)

International (IPC 1-7): A23L1/39 A23L1/40 A23P1/02

CPC: A23L1/0026 A23L1/22 A23L1/39 A23L1/40

European: A23L1/00P2D A23L1/22 A23L1/39 A23L1/40



Family:

Publication number	Publication date	Application number	Application date
AT503395 E	20110415	AT20020716749T	20020208
AU2002247700 AA	20020919	AU20020247700	20020208
AU2008201631 AA	20080501	AU20080201631	20080411
AU2008201631 BB	20091008	AU20080201631	20080411
BG108138 A	20050131	BG20030108138	20030828
BG66337 B1	20130731	BG20030108138	20030828
BRPI0207942 A	20040302	BR2002PI07942	20020208
CA2441956 AA	20030923	CA20022441956	20020208
CN100421580 C	20081001	CN20028009433	20020208
CN1507325 A	20040623	CN20028009433	20020208
CZ20032715 A3	20031217	CZ20030002715	20020208
DE20103914 U1	20020711	DE20012003914U	20010306
DE50214989 D1	20110512	DE20025014989	20020208
EP1370160 A1	20031217	EP20020716749	20020208
EP1370160 B1	20110330	EP20020716749	20020208
IL157758 A0	20040328	IL20020157758	20020208
IL157758 A1	20060221	IL20030157758	20030904
MXPA03007995 A1	20031204	MX2003PA07995	20030905
PL363626 A1	20041129	PL20020363626	20020208
RU2003129520 A	20050420	RU20030129520	20020208
RU2302175 C2	20070710	RU20030129520	20020208
UA30928 U	20080325	UA20031008982U	20020208
WO02069742 A1	20020912	WO2002EP01346	20020208

Priority:

DE20012003914U 20010306 AU20020247700 20020208 WO2002EP01346 20020208
DE20025014989 20020208 AU20080201631 20080411

Assignee(s): (std): DES PRODUITS NESTLE SOC SA ; LENOBLE HENRI PIERRE ; NESTLE SA ; PRODUITS NESTEL S A SOC D ; SOCIETE DES PRODUITS NESTLE S A

Assignee(s): SOCIETE DES PRODUITS NESTLE S A VEVEY ; SOS ETE DE PRODJUI NESTLE S A ; DES PRODUITS NESTLE SA SOC ; SOCIETE DES PRODUITS NESTEL S A

Inventor(s): (std): HP LENOBLE ; LENOBL ANRI P ER ; LENOBLE H P ; LENOBLE HENRI PIERRE ; LENOBLE HERI PIERRE

Inventor(s): LENOBL' ANRI-P'ER ; H P LENOBLE ; HENRI PIERRE LENOBLE ; HERI PIERRE LENOBLE

Agent(s): THOMAS ALAIN

Designated states: AE AG AL AM AT AU AZ BA BB BE BF BG BJ BR BY BZ CA CF CG CH CI CM CN CO CR CU CY CZ DE DK DM DZ EC EE ES FI FR GA GB GD GE GH GM GN GQ GR GW HR HU ID IE IL IN IS IT JP KE KG KP KR KZ LC LI LK LR LS LT LU LV MA MC MD MG MK ML MN MR MW MX MZ NE NL NO NZ OM PH PL PT RO RU SD SE SG SI SK SL SN SZ TD TG TJ TM TN TR TT TZ UA UG US UZ VN YU ZA ZM ZW

Title: SOFT BOUILLON TABLET

Abstract: A soft bouillon tablet which comprises only or mainly oil and no or little fat beside non fat conventional bouillon ingredients.

Classifications:

International (IPC 8-9): A23L1/00 A23L1/22 A23L1/237 A23L1/29 A23L1/314 A23L1/325 A23L1/39 A23L1/40 A23P1/02 A23P1/08 (Advanced/Invention)

International (IPC 1-7): A23L1/00 A23L1/22 A23L1/237 A23L1/29 A23L1/314 A23L1/325 A23L1/39 A23L1/40 A23P1/02 A23P1/08

CPC: A23L1/40

European: A23L1/40

Family:

Publication number	Publication date	Application number	Application date
AR034536 AA	20040225	AR2002P102325	20020620
AT362329 E	20070615	AT20020754595T	20020527
AU2002321043 AA	20030108	AU20020321043	20020527
AU2002321043 BB	20070607	AU20020321043	20020527
BRPI0210543 A	20040622	BR2002PI10543	20020527
CA2449882 AA	20031205	CA20022449882	20020527
CA2449882 C	20110712	CA20022449882	20020527
CN1518419 A	20040804	CN20028012399	20020527
CZ20033524 A3	20040414	CZ20030003524	20020527
DE60220163 D1	20070628	DE20026020163	20020527
DE60220163 T2	20080214	DE20026020163T	20020527
DOP20020425 A	20031230	DO20020000425	20020625
EP1269863 A1	20030102	EP20010202381	20010620
EP1401296 A1	20040331	EP20020754595	20020527
EP1401296 B1	20070516	EP20020754595	20020527
ES2284903 T3	20071116	ES20020754595T	20020527
GT200200128 A	20030619	GT20020000128	20020620
IL159090 A0	20040512	IL20020159090	20020527
IL159090 A1	20060705	IL20030159090	20031127
NZ530195 A	20060428	NZ20020530195	20020527
PE00702003 A1	20030314	PE20020000533	20020620
PL208843 B1	20110630	PL20020367220	20020527
PL367220 A1	20050221	PL20020367220	20020527
PT1401296 T	20070531	PT20020754595T	20020527
RU2004101295 A	20050510	RU20040101295	20020527
RU2308206 C2	20071020	RU20040101295	20020527
TH61172 A	20040317	TH20021002237	20020618
WO03000077 A1	20030103	WO2002EP05833	20020527

Priority:

EP20010202381 20010620 EP20020754595 20020527 WO2002EP05833 20020527

Assignee(s): (std): NESTLE SA ; FROEHLICH MARKUS ; PALZER STEFAN ; RUEEGER MAX ; SCHMUTZ WALTER ; DES PRODUITS NESTLE SOC SA ; BRUSONI GIANLUCA ; SOLTERMANN PATRICK

Assignee(s): SOS ETE DE PRODUI NESTLE S A ; DES PRODUITS NESTLE S A SOC ; DES PRODUITS NESTLE S SOC ; DES PRODUITS NESTLE SA SOC ; SOCIETE DES PRODUITS NESTLE S ; RUEGER MAX

Inventor(s): (std): FROEHLICH M ; FROEHLICH MARCUS ; FROEHLICH MARKUS ; M FROEHLICH ; M RSEGER ; BRUSONI GIANLUCA ; PALZER S ; PALZER STEFAN ; RSEGER M ; RUEEGER MAX ; RUEGER MAX ; S PALZER ; SCHMUTZ WALTER ; SOLTERMANN PATRICK ; ZOL Terman PATRIK ; SHMUTTS VAL TER ; RJUEGER MAKs ; PAL TSER SHTEFAN ; FRELIKH MARKUS ; BRUZIONI G JANLUKA

Inventor(s): STEFAN PALZER ; WALTER SCHMUTZ ; ZOL'TERMAN PATRIK ; SHMUTTS VAL'TER ; PATRICK SOLTERMANN ; BRUZIONI G'JANLUKA ; MARCUS FROEHLICH ; MARKUS FROEHLICH ; MAX RUEEGER ; MAX RUEGER ; PAL'TSER SHTEFAN ; GIANLUCA BRUSONI

Designated states: AE AG AL AM AT AU AZ BA BB BE BF BG BJ BR BY BZ CA CF CG CH CI CM CN CO CR CU CY CZ DE DK DM DZ EC EE ES FI FR GA GB GD GE GH GM GN GQ GR GW HR HU ID IE IL IN IS IT JP KE KG KP KR KZ LC LI LK LR LS LT LU LV MA MC MD MG MK ML MN MR MW MX MZ NE NL NO NZ OM PH PL PT RO RU SD SE SG SI SK SL SN SZ TD TG TJ TM TN TR TT TZ UA UG US UZ VN YU ZA ZM ZW

Title: PROBIOTIC DELIVERY SYSTEM

Abstract: Source: US2005153018A The present invention relates to a probiotic delivery system that is preferably added to a food product. In particular, the invention shows that compacted pellets having a volume of at least 0.02 cm³, that comprise, besides viable micro-organisms, arbitrary or eligible components, such as fillers, binder, plasticizer, other functional ingredients and a coating may be added to semi-moist, moist or semi-dry products. The micro-organisms remain viable for a longer time than commercially obtainable preparations of probiotics.

Classifications:

International (IPC 8-9): A23K1/00 A23K1/16 A23K1/18 A23K1/20 A23L1/00 A23L1/03 A23L1/28 A23L1/30 A61K35/66 A61K35/74 A61K9/16 A61K9/54 A61P1/12 A61P1/14 A61P3/06 A61P31/00 A61P37/02 C12N1/04 C12N11/00 C12N11/04 (Advanced/Invention)

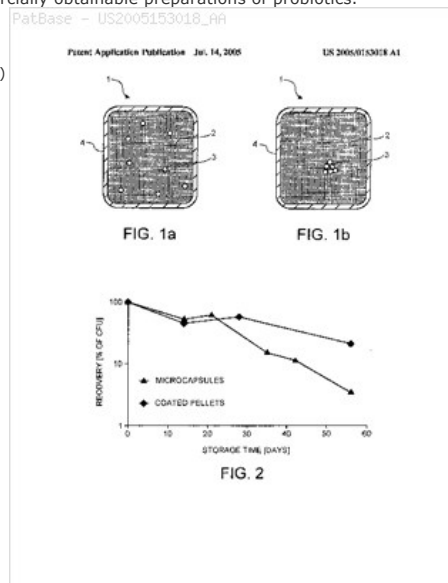
International (IPC 1-7): A23K1/00 A23K1/03 A23K1/16 A23K1/18 A23K1/20 A23L1/03 A23L1/28 A23L1/30 A61K35/66 A61K35/74 A61K9/16 A61P1/12 A61P1/14 A61P3/06 A61P31/00 A61P37/02 C12N1/04

CPC: C12N1/04 A23K1/002 A23K1/003 A23K1/004 A23K1/009 A23L1/0029 A23L1/0055 A23L1/0345 A23L1/3014 A23V2002/00 A61K35/741

European: A23K1/00B1 A23K1/00B2 A23K1/00B3 A23K1/00C2B A23L1/00P4

A23L1/00P8B8 A23L1/03M A23L1/30M A61K35/74 A61K35/74A C12N1/04 K23V2/00

US: 426/61 426/61P

**Family:**

Publication number	Publication date	Application number	Application date
AU2003218748 AA	20030922	AU20030218748	20030312
AU2003218748 BB	20071101	AU20030218748	20030312
BRPI0308586 A	20050111	BR2003PI08586	20030312
CA2478553 AA	20040902	CA20032478553	20030312
CA2478553 C	20120515	CA20032478553	20030312
CN100334964 C	20070905	CN20038005801	20030312
CN1642437 A	20050720	CN20038005801	20030312
DE60307993 D1	20061012	DE20036007993	20030312
DE60307993 T2	20070222	DE20036007993T	20030312
EP1344458 A1	20030917	EP20020005607	20020312
EP1482811 A1	20041208	EP20030711996	20030312
EP1482811 B1	20060830	EP20030711996	20030312
ES2269993 T3	20070401	ES20030711996T	20030312
IN03105DN2004 A	20091204	IN2004DN03105	20041011
IN250290 B	20111223	IN2004DN03105	20041011
JP2005519600 T2	20050707	JP20030573962T	20030312
JP2011055832 A2	20110324	JP20100204628	20100913
MXPA04008814 A1	20050908	MX2004PA08814	20040910
RU2004130318 A	20050610	RU20040130318	20030312
RU2323586 C2	20080510	RU20040130318	20030312
US2005153018 AA	20050714	US20030507359	20030312
WO03075676 A1	20030918	WO2003EP02597	20030312
ZA200408199 A	20051011	ZA20040008199	20041011

Priority:

EP20020005607 20020312 EP20030711996 20030312 WO2003EP02597 20030312
JP20030573962 20030312

Assignee(s): (std): NESTLE SA ; UBBINK JOHAN BERNARD ; SCHAEER ZAMMARETTI PRISCA ; NESTEC SA ; SOCIETE DES PRODUITS NESTLE S A ; CAVADINI CHRISTOPH ; DES PRODUITS NESTLE SOC SA

Assignee(s): NESTEC S A ; DES PRODUITS NESTLE S A SOC ; DES PRODUITS NESTLE SA SOC ; SOS ETE DE PRODUITS NESTLE S A ; SOC DES PRODUITS NESTLE SA ; SOS ETE DE PRODUITS NESTLE SA

Inventor(s): (std): BERNARD UBBINK JOHAN ; CAVADINI CHRISTOPH ; CHRISTOPH CAVADINI ; CHRISTOPH UBBINK JOHAN BERNARD ; JOHAN BERNARD UBBINK ; KAVADINI KRISTOF ; PRISCA SCHAEER ZAMMARETTI ; SCHAEER ZAMMARETTI PRISCA ; SHAEHR ZAMARETTI PRISKA ; UBBINK JOKHAN BERNARD ; UBBINK JOHAN BERNARD ; UBBINK JOHAN B

Inventor(s): UBBINK JOHAN BERNARD SCHAEER ZAMMARETTI PRISCA ; UNBINK JOHAN ; UNBINK JOHAN BERNARD ; UBBINK JOHAN BERNARD SCHAEER ZAMMARETTI PRISCA CAVADINI CHRISTOPH ; SHAEHR-ZAMARETTI PRISKA ; UBBINK JOHAN

Agent(s): SANJAY AGRAWAL

Designated states: AE AG AL AM AT AU AZ BA BB BE BF BG BJ BR BY BZ CA CF CG CH CI CM CN CO CR CU CY CZ DE DK DM DZ EC EE ES FI FR GA GB GD GE GH GM GN GQ GR GW HR HU ID IE IL IN IS IT JP KE KG KP KR KZ LC LI LK LR LS LT LU LV MA MC MD MG MK ML MN MR MW MX MZ NE NL NO NZ OM PH PL PT RO RU SD SE SG SI SK SL SN SZ TD TG TJ TM TN TR TT TZ UA UG US UZ VN YU ZA ZM ZW

Title: HARD BOUILLON TABLET OR CUBE

Abstract: A hard bouillon tablet or cube which comprises only or mainly liquid end oil and no or little fat besides non fat conventional bouillon ingredients.

Classifications:

International (IPC 8-9): A23L1/00 A23L1/30 A23L1/40 (Advanced/Invention); A23L (Subclass/Non-invention)

International (IPC 1-7): A23L1/00 A23L1/22 A23L1/40

CPC: A23L1/0026 A23L1/3006 A23L1/40 A23V2002/00

European: A23L1/00P2D A23L1/30C A23L1/40 K23V2/00

Family:

Publication number	Publication date	Application number	Application date
AP200503321 A0	20050630	AP20050003321	20021202
AP2101 A	20100204	AP20050003321	20021202
AU2002358589 AA	20040623	AU20020358589	20021202
WO04049831 A1	20040617	WO2002EP13683	20021202

Priority:

AP20050003321 20021202 WO2002EP13683 20021202

Assignee(s): (std): NESTEC SA

Assignee(s): NESTEC S A

Inventor(s): (std): GNOU JEAN ; KOUADIO MARTIN ; MOUNIER GEORGES ; PALZER STEFAN ; SORO MAMADOU ; PALZER STEPHAN ; GNOU JEAN ET AL

Inventor(s): GEORGES MOUNIER ; STEFAN PALZER ; MAMADOU SORO ; MARTIN KOUADIO ; JEAN GNOU

Designated states: AE AG AL AM AT AU AZ BA BB BE BF BG BJ BR BY BZ CA CF CG CH CI CM CN CO CR CU CY CZ DE DK DM DZ EC EE ES FI FR GA GB GD GE GH GM GN GQ GR GW HR HU ID IE IL IN IS IT JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MC MD MG MK ML MN MR MW MX MZ NE NL NO NZ OM PH PL PT RO RU SD SE SG SI SK SL SN SZ TD TG TJ TM TN TR TT TZ UA UG UZ VN YU ZA ZM ZW

Title: SOLID CONCENTRATE FOR BROTHS, BOUILLONS, SOUPS, SAUCES, (SIDE)DISHES, GARNISHING OR SEASONING AND PROCESS FOR PREPARING THE SAME

Abstract: Source: US2007134397A Concentrates for bouillons, broths, soups, sauces, gravies, dishes, side dishes, or for use as garnishing or seasoning containing dry herbs and/or vegetables, which concentrate is in the form of a solid body or bodies having a weight of 2-250 g, in particular a cube, block, or tablet shape, which yield a soup, sauce etcetera upon dilution with (hot) water or an aqueous liquid, or can be used as a (side)dish, seasoning or garnishing, and wherein the dry herbs and/or vegetables are present in or with the concentrate as one or more compressed particles or layers.

Classifications:
International (IPC 8-9): A23L1/00 A23L1/212 A23L1/40 A23P1/02 A23P1/08 (Advanced/Invention); A23L1/00 A23L1/212 A23L1/39 A23P1/02 A23P1/08 (Core/Invention)
International (IPC 1-7): A23L1/212 A23L1/40 A23P1/02
CPC: A23L1/0026 A23L1/2125 A23L1/40
European: A23L1/00P2D A23L1/212C2 A23L1/40
US: 426/589

Family:

Publication number	Publication date	Application number	Application date
AR056848 AA	20071024	AR2006P105434	20061211
EP1965662 A1	20080910	EP20060829106	20061122
EP1965662 B1	20150304	EP20060829106	20061122
US2007134397 AA	20070614	US20060633809	20061205
WO07068332 A1	20070621	WO2006EP11214	20061122

Priority:
EP20050077840 20051212 WO2006EP11214 20061122 EP20060829106 20061122

Assignee(s): (std): CONOPCO INC D B A UNILEVER ; CONOPCO INC DBA UNILEVER ; EPPLER WOLFGANG MICHAEL ; MUELLER ANDREA SILVIA ; MELWITZ DIETER WERNER ; UNILEVER NV ; LEVER HINDUSTAN LTD ; MAHUT CLAUDE GERARD JEAN BERNA ; UNILEVER PLC

Assignee(s): HINDUSTAN LEVER LIMITED ; HINDUSTAN LEVER LTD ; MAHUT CLAUDE GERARD JEAN BERNARD ; UNILEVER N V ; CONOPCO INC

Inventor(s): (std): MELWITZ DIETER W ; MELWITZ DIETER WERNER ; MUELLER ANDREA S ; MUELLER ANDREA SILVIA ; EPPLER CECIL MARK ; EPPLER WOLFGANG M ; EPPLER WOLFGANG MICHAEL ; MAHUT CLAUDE GERARD JEAN BERNA ; MAHUT CLAUDE GERARD JEAN BERNARD ; MAHUT JEAN BERNARD G C ; MAHUT JEAN BERNARD GERARD CLAU

Inventor(s): MAHUT JEAN BERNARD GERARD CLAUDE ; MAHUT JEAN BERNARD GERARD C ; MUELLER ANDREA ; MELWITZ DIETER ; MAHUT JEAN BERNARD ; EPPLER WOLFGANG

Agent(s): WURFBAIN GILLES L; REIJNS TIEMEN GEERT PIETER; UNILEVER INTELLECTUAL PROPERTY GROUP

Designated states: AE AG AL AM AT AU AZ BA BB BE BF BG BJ BR BW BY BZ CA CF CG CH CI CM CN CO CR CU CY CZ DE DK DM DZ EC EE EG ES FI FR GA GB GD GE GH GM GN GQ GR GT GW HN HR HU ID IE IL IN IS IT JP KE KG KM KN KP KR KZ LA LC LI LK LR LS LT LU LV LY MA MC MD MG MK ML MN MR MW MX MY MZ NA NE NG NI NL NO NZ OM PG PH PL PT RO RS RU SC SD SE SG SI SK SL SM SN SV SY SZ TD TG TJ TM TN TR TT TZ UA UG US UZ VC VN ZA ZM ZW

Title: A BOUILLON AND/OR SEASONING TABLET CONTAINING CEREAL, VEGETABLE AND/OR FRUIT FIBERS

Abstract: The present invention concerns a bouillon and/or seasoning tablet and/or cube, which comprises, in total percent in weight, 0.5-10 percent oil and/or 0-5 percent fat, binders, salt, between 2 and 3 percent of total water, and between 0.5 and 8 percent of cereal, vegetable or fruit fibers, as well as optionally sugar, spices, flavors, taste enhancers, dehydrated vegetables, herb leaves and/or plant extracts.

Classifications:
International (IPC 8-9): A23L1/0534 A23L1/22 A23L1/40 (Advanced/Invention);
A23L1/052 A23L1/39 (Core/Invention);
A23L (Subclass/Invention)
CPC: A23L1/40 A23L1/0534 A23V2002/00 A23V2250/5116
European: A23L1/0534 A23L1/40 K23V2/00 K23V250/5116

Family:

Publication number	Publication date	Application number	Application date
AU2007209344 AA	20070802	AU20070209344	20070124
AU2007209344 BB	20121220	AU20070209344	20070124
BRPI0706727 A2	20110405	BR2007PI06727	20070121
CA2637086 AA	20080714	CA20072637086	20070124
CA2637086 C	20140610	CA20072637086	20070124
CN101374427 A	20090225	CN200780003222	20070124
CN101374427 B	20130313	CN200780003222	20070124
EP1820409 A1	20070822	EP20060001417	20060124
EP1978831 A1	20081015	EP20070704112	20070124
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Priority:
EP20060001417 20060124 WO2007EP50680 20070124 EP20070704112 20070124

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Designated states: AE AG AL AM AT AU AZ BA BB BE BF BG BJ BR BW BY BZ CA CF CG CH CI CM CN CO CR CU CY CZ DE DK DM DZ EC EE EG ES FI FR GA GB GD GE GH GM GN GQ GR GT GW HN HR HU ID IE IL IN IS IT JP KE KG KM KN KP KR KZ LA LC LI LK LR LS LT LU LV LY MA MC MD MG MK ML MN MR MW MX MY MZ NA NE NG NI NL NO NZ OM PG PH PL PT RO RS RU SC SD SE SG SI SK SL SM SN SV SY SZ TD TG TJ TM TN TR TT TZ UA UG US UZ VC VN YU ZA ZM ZW

Title: PACKAGING COMPRISING ONE OR MORE OF COATED SOLID CONCENTRATES FOR PREPARING A BOUILLON SOUP, SAUCE OR GRAVY OR FOR USE AS A SEASONING AND PROCESS FOR PREPARING THE SAME

Abstract: ABSTRACT Container comprising a one or more of concentrate articles for preparing a bouillon, soup, sauce, roux, gravy or for use as a seasoning, the concentrate articles comprising a solid core material covered by an outer layer completely enclosing the core material, wherein the core is prepared by roller compacting. The invention also relates to a process of obtaining such concentrates.

Classifications:

International (IPC 8-9): A23L1/00 A23L1/40 B65D85/64 (Advanced/Invention);
B65D85/64 (Advanced/Non-invention);
A23L1/00 A23L1/39 B65D85/64 (Core/Invention);
B65D85/64 (Core/Non-invention)
CPC: A23L1/0047 A23L1/40
European: A23L1/00P8B A23L1/40

Family:

Publication number	Publication date	Application number	Application date
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Priority:

WO2006EP02298 20060310 RU20080140180 20060310

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Inventor(s): GEORG ACHTERKAMP ; KARL WALTER FERDINAND SCHARF ; SHARF KARL VAL'TER FERDINAND ; K W F SCHARF ; G ACHTERKAMP

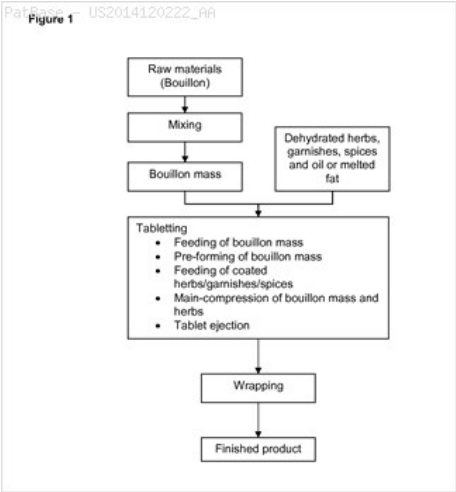
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Designated states: AE AG AL AM AT AU AZ BA BB BE BF BG BJ BR BW BY BZ CA CF CG CH CI CM CN CO CR CU CY CZ DE DK DM DZ EC EE EG ES FI FR GA GB GD GE GH GM GN GQ GR GW HR HU ID IE IL IN IS IT JP KE KG KM KN KP KR KZ LC LI LK LR LS LT LU LV LY MA MC MD MG MK ML MN MR MW MX MZ NA NE NG NI NL NO NZ OM PG PH PL PT RO RU SC SD SE SG SI SK SL SM SN SY SZ TD TG TJ TM TN TR TT TZ UA UG US UZ VC VN YU ZA ZM ZW

Title: BOUILLON TABLET PROCESS

Abstract: Source: US2014120222A A process for preparing a bouillon tablet having two or more layers comprising the steps (a) forming a first layer of the tablet comprising a mixture of two or more of salt, taste enhancer, sugar, and fat (or oil), (b) forming a second layer of the tablet by adding to the first layer a mixture comprising one or more of herbs, garnishes, vegetable pieces, and spices; and (a) compressing the two layers to form the tablet, where the tablet is formed using a rotary double-punch press.

Classifications:
International (IPC 8-9): A23L A23L1/00 A23L1/22 A23L1/221 A23L1/40 (Advanced/Invention)
CPC: A23L1/0026 A23L1/40
US: 426/285 426/589



Family:

Publication number	Publication date	Application number	Application date
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Priority:
EP20110170231 20110616 EP20110070231 20110616 WO2012EP60193 20120530
EP20120725376 20120530

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Designated states: AE AG AL AM AO AT AU AZ BA BB BE BF BG BH BJ BR BW BY BZ CA CF CG CH CI CL CM CN CO CR CU CY CZ DE DK DM DO DZ EC EE EG ES FI FR GA GB GD GE GH GM GN GQ GR GT GW HN HR HU ID IE IL IN IS IT JP KE KG KM KN KP KR KZ LA LC LI LK LR LS LT LU LV LY MA MC MD ME MG MK ML MN MR MT MW MX MY MZ NA NE NG NI NL NO NZ OM PE PG PH PL PT QA RO RS RU RW SC SD SE SG SI SK SL SM SN ST SV SY SZ TD TG TH TJ TM TN TR TT TZ UA UG US UZ VC VN ZA ZM ZW