Clarke, Modet
COLOMBIA

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
DIVISION DE NUEVAS CREACIONES
E. S. D.

Re: Expediente No. **05-091.249**
Solicitud de patente a nombre de **Givaudan S.A.**

Yo, JIMENA ESCOBAR URIBE, identificada como aparece al pie de mi firma, domiciliada en la Carrera 11 No. 86-53, piso 6, obrando como apoderada de la sociedad **Givaudan S.A.** domiciliada en Vernier, Suiza, solicitante de la patente de la referencia, en respuesta al oficio No. 13293 notificado por fijación en lista el 17 de octubre de 2008, relacionado con el concepto técnico de fondo No. 2596, para el cual se solicitó prórroga, me permito manifestar:

1. Con fundamento en la facultad que confiere el artículo 34 de la Decisión 486 al solicitante para modificar la solicitud de patente en cualquier momento del trámite, presento junto con este memorial nuevo capítulo reivindicatorio en el cual se realizaron las siguientes modificaciones:

1.1 Como primera medida, se modificó la reivindicación independiente 1, con el fin de incluir algunas características descritas en la descripción y en las antiguas reivindicaciones 4 y 5.

1.2 De forma complementaria, me permito manifestar que se limitó el alcance de la invención, toda vez que se precisaron en la reivindicación 1 los rangos para la proporción en peso de la sal soluble en agua y el portador que se adicionan para preparar la composición.

1.3 Por otro lado, se modificó la antigua reivindicación 5 (nueva reivindicación 4), con el fin de limitar el alcance de la invención y los porcentajes de componentes presentes en la composición.

1.4 Además, se incluyeron nuevas características en la nueva reivindicación 4 (antigua reivindicación 5), donde se define el portador particulado.

1.5 Se eliminó la antigua reivindicación 4, ya que las características allí definidas se encuentran en las nuevas reivindicaciones 1 y 4.

1.6 Finalmente, se eliminó la antigua reivindicación 8 por considerarse que dicha cláusula se refiere a un uso.

En cuanto a estas modificaciones me permito decir que las mismas no amplían de ninguna manera la materia objeto de la presente solicitud y se realizaron con el ánimo de superar las objeciones de claridad, reivindicaciones relativas a un uso o a un segundo uso, novedad y de nivel inventivo planteadas por el examinador en su concepto técnico.

Para este efecto, me permito acompañar el recibo de pago No. 09 – 8,502 de febrero 3 de 2009, con los cuales queda cubierta la tasa correspondiente a la modificación de una solicitud de patente en cuanto a su capítulo reivindicatorio.

2. Claridad de la solicitud

Respecto a este punto, el solicitante manifiesta que mediante las modificaciones realizadas en el capítulo reivindicatorio, quedan subsanadas la totalidad de las objeciones, de modo tal que las nuevas reivindicaciones proporcionan claridad y concisión a la invención descrita, siguiendo las recomendaciones realizadas por el examinador en su concepto técnico.

3. Reivindicaciones relativas a un uso o a un segundo uso

Con relación a esta objeción planteada por el examinador, el solicitante manifiesta que mediante las modificaciones realizadas a las reivindicaciones, específicamente las mencionadas en el punto 1.6 del presente memorial, queda subsanada la objeción en referencia a las supuestas reivindicaciones relativas a un uso o segundo uso.

4. Novedad

El examinador establece en su concepto técnico No. 2596 que las antiguas reivindicaciones 1 a 8 de la presente solicitud carecen de novedad a la luz de los documentos US 6426325 (D1), ES 2222238 (D2), US 5656584 (D3) y US 4873000 (D4).

En este sentido, me permito decir que el artículo 16 de la Decisión 486 de la Comisión de la Comunidad Andina establece que la invención se considera como nueva cuando no está comprendida en el estado de la técnica. Si de acuerdo con el espíritu de la norma legal indicada, la invención no se considera como nueva si está "comprendida" en el estado de la técnica, dicha norma se está refiriendo, naturalmente, a la totalidad del invento y no a alguno o a algunos de los componentes. Para que la invención pierda su calidad de novedosa, es claro que ella, TODA ELLA, ha de estar contenida en el "*conjunto de conocimientos existentes que son aplicables al proceso industrial*" como define el estado de la técnica el Tribunal de justicia del Acuerdo de Cartagena (Proceso No. 6-IP-89, sentencia de 31 de octubre de 1989, publicada en Jurisprudencia del Tribunal de Justicia del Acuerdo

de Cartagena, Tomo 11, BID-INTAL, Buenos Aires, 1994, página 60).

Esto se conoce como NOVEDAD ABSOLUTA, de conformidad con lo cual para desvirtuar el requisito de novedad de una invención será necesario que lo reivindicado por la invención bajo estudio corresponda de forma total con lo revelado en un documento o anterioridad señalada, conclusión a la que se llega después de un análisis exhaustivo mediante una comparación entre el capítulo reivindicatorio de la invención en estudio y la divulgación de las referencias D1, D2, D3 y D4.

Ahora bien, si un determinado elemento, condición o circunstancia de la nueva creación ya hace parte del estado de la técnica no quiere ello decir, desde luego, que la invención deba ser rechazada por carecer del requisito de novedad. No sin razón ha dicho el mencionado Tribunal: "*hay que advertir que toda creación es en cierta forma, **producto del estado de la técnica existente**, en el sentido de que el inventor ha de tener en cuenta un conjunto de conocimientos técnicos que le permitan **crear nuevos procedimientos aplicables a la industria***" (Ibídem, páginas 53 y 54) (el subrayado es nuestro).

Por lo tanto, como mínimo la novedad debe contemplar los siguientes aspectos:

-Que la invención en estudio esté contenida de forma TOTAL y ABSOLUTA en el documento que se menciona como anterioridad o de **forma independiente** en los documentos que se mencionan, si son varios.

-Que la invención en estudio puede partir de conocimientos conocidos, caso en el cual habrá que hacer la comparación no de lo conocido sino de lo inédito y novedoso, teniendo que analizarse el valor agregado que aporta la invención con sus nuevas características al campo técnico al cual pertenece la invención en estudio.

Aplicando lo anterior al caso concreto, para que las razones que aduce ese despacho para desvirtuar la novedad fuesen ciertas, sería forzoso que se demostrara que la invención de mi mandante esté comprendida en el estado de la técnica de forma **TOTAL**, lo cual no sucede con la materia reclamada en las nuevas reivindicaciones.

Así las cosas, es deseo del solicitante manifestar que mediante las modificaciones realizadas a las reivindicaciones de la presente solicitud, tanto el método descrito en las reivindicaciones 1 a 3, como la composición reclamada en las reivindicaciones 4 a 6 son novedosos, toda vez que ninguno de los documentos mencionados como anterioridades menciona específicamente que la fragancia que se adiciona se encuentre en la forma de un líquido o una solución de solvente orgánico, ni tampoco sugiere que la cantidad de sal soluble en agua sea de 60% a

85% en peso y la cantidad de portador en la composición oscila entre 5% y 20% en peso de la composición, lo que representa una ventaja significativa sobre lo que existía en el estado del arte en el momento de la presentación de la solicitud prioritaria.

Adicionalmente, es importante resaltar el hecho que los documentos mencionados por el examinador no terminan siendo completamente relevantes para la presente solicitud, toda vez que el documento D1 se refiere a un substrato particulado inorgánico sólido **no absorbente**, lo cual difiere en su totalidad de la presente invención teniendo en cuenta que la composición reclamada comprende sílice porosa fina. Basándose en este dato, una persona versada en la materia encontraría y reconocería que la presente invención es diferente.

Por otro lado, el documento D2 menciona que "*La presente invención proporciona en uno de sus aspectos un producto de cuidado doméstico que comprende una partícula de fragancia en el que la partícula de fragancia comprende una composición de fragancia y al menos un* ***polímero de silicona*** *que tiene un punto de fusión de al menos 10°C (...)*" (el subrayado es nuestro). Siendo así, se puede notar que la partícula definida en dicho documento es un polímero de silicona, lo cual corresponde a una tecnología completamente diferente a la que se desea proteger con la solicitud en estudio, que se refiere a una partícula compuesta por una sal de un metal álcali o alcalino térreo soluble en agua y un portador de sílice porosa.

Ahora bien, con relación al documento D3, éste define que "*El proceso esencialmente comprende los pasos de mezclar uniformemente un* ***material encapsulado****, preferiblemente un material de carbohidrato vidrioso, con un material portador poroso, preferiblemente cargado con un perfume, y luego compactar la mezcla en aglomerados*" (el subrayado es nuestro). En este sentido, se puede ver claramente que dicho documento necesita de un material encapsulado, hecho que no se presenta en la solicitud en estudio, ya que dicho tipo de materiales no son utilizados para lograr la composición y el método reclamados en la presente invención.

Finalmente, el documento US 4873000 (D4) se refiere a una composición completamente diferente, no sólo porque dicha composición debe comprender arcilla de silicato de aluminio, sino porque también es necesario que contenga un agente aglomerante. Contario a ello, es importante tener en cuenta que la presente invención no menciona en ninguna parte que dichos componentes sean necesarios para obtener la fragancia o composición que se desea proteger.

Por lo tanto, las nuevas reivindicaciones 1 a 6, cumplen con lo establecido en el artículo 16 de la Decisión 486, ya que la materia allí descrita no ha sido divulgada de forma completa y no se ha desvirtuado la NOVEDAD ABSOLUTA de la presente solicitud.

5. Nivel Inventivo

El artículo 18 de la Decisión 486 determina que "*una invención tiene nivel inventivo, si para una* ***persona del oficio normalmente versada en la materia técnica*** *correspondiente, esa invención no hubiese resultado obvia ni se hubiese derivado de manera evidente del estado de la técnica*". (El subrayado es nuestro).

Lo anterior significa que el análisis de nivel inventivo no es una comparación de los objetos de las anterioridades y la solicitud en estudio. El análisis de nivel inventivo debe demostrar la obviedad de la invención y cómo ésta no contiene un salto cualitativo, que demuestre su altura inventiva.

Así las cosas, no es clara la discusión del examinador en relación de cómo la presente invención carece de nivel inventivo. Si bien afirma que para una persona versada en la materia es obvia la invención, no sustenta ni prueba ni motiva la manera como esta invención resulta obvia. No obstante, mi mandante ilustra a continuación con suficientes argumentos cómo esta solicitud NO resulta obvia para una persona versada en la materia y además, representa un avance a lo que existía en el estado del arte en el momento de la presentación de la solicitud prioritaria.

En este orden de ideas, es deseo del solicitante manifestar que el capítulo descriptivo de la solicitud en estudio menciona un problema que existía en el estado del arte al momento de la presentación de la solicitud prioritaria, el cual fue definido en los siguientes términos: "*muchos productos para el lavado contienen tensioactivos, los cuales forman micelios en agua, y, como muchas fragancias son hidrobóficos, tienden a migrar a los micelios, en lugar de depositarse en el sustrato*".

En este sentido, la presente solicitud proporciona una solución a dicho problema que se encontraba en el arte previo, toda vez que la presente invención divulga "*un método de preparación de una composición que proporciona fragancia sólida de flujo libre, que consiste esencialmente de la adición de una fragancia a un material portador particulado en la presencia de una sal soluble en agua de un metal álcali o alcalino térreo*" y, adicionalmente, "*Las composiciones de la presente invención difieren considerablemente de composiciones conocidas, las cuales tienen altas proporciones de portador particulado (usualmente en exceso de 40% en peso) y altas cargas de perfume (típicamente de 20-50% en peso de la composición total)*".

En consecuencia, es posible establecer que el método y la composición descritas en las nuevas reivindicaciones 1 a 6 de la presente solicitud representan una solución a las desventajas o problemas que existían en el estado del arte al momento de la presentación de la solicitud prioritaria.

En este orden de ideas, se puede manifestar que la presente invención cumple

con el requisito de nivel inventivo tal y como se encuentra establecido en el artículo 18 de la Decisión 486.

En resumen, me permito decir que gracias a las modificaciones expuestas en el punto 1 de este memorial y los argumentos expuestos anteriormente, la presente solicitud cumple cabalmente con los requisitos de claridad, novedad y nivel inventivo respecto al método para la preparación de una composición que proporciona fragancia expuesto en el nuevo capítulo reivindicatorio. Por lo tanto, solicito comedidamente a ese despacho se sirva reconocer estos requisitos y conceder el privilegio de patente de invención a la solicitud en estudio.

En consecuencia, solicitamos a ese despacho pronunciarse en relación con todos los argumentos presentados en este memorial dando estricta aplicación al principio del debido proceso consagrado en el artículo 29 de la Constitución Nacional. De igual forma, si el examinador considera que subsisten razones para objetar la presente invención ruego expedir otro examen de fondo con los debidos motivos y razones que sustenten su objeción con el fin de que mi mandante tenga la posibilidad de exponer sus argumentos con base en la interpretación de los artículos 45 de la Decisión 486 -el cual otorga la facultad al examinador para emitir los exámenes de fondo-, 35 y 59 del Código Contencioso Administrativo que señala que: *"habiéndose dado oportunidad a los interesados para expresar sus opiniones, y con base en las pruebas e informes disponibles, se tomará la decisión que será motivada al menos en forma sumaria si afecta a particulares"* y *"(..) deberá proferirse la decisión definitiva. Esta se motivará en sus aspectos de hecho y de derecho, y en los de conveniencia si es el caso"*, respectivamente (el subrayado es nuestro).

Señor Superintendente,

JIMENA ESCOBAR URIBE
C.C.39.779.452 de Usaquén
T.P.No. 68228
Còd.5376

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO

NIT : 800176089-2

RECIBO OFICIAL DE CAJA : 09 - 8,502
FECHA : FEBRERO 3 DE 2009

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
No. 05-091249- -00006-0000
Fecha: 2009-02-12 14:59:54 Dep: 2020 NULIDAD RELACIONE
Tra: 11 PATENTE(YII Lve: 378 PASENACIONALI
Act: 444 RESPUESTA RECOUE Folios: 9

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

DEPOSITANTE	TIPO PAGO	BANCO	CUENTA	No. PAGO	FECHA PGO	Vr. PAGO
CLARKE MODET Y CO. COLOMB	CONSIGNACION	BANCO DE BOGOTA	062754387	108004750	02/02/2009	31,570,000.00

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

CANT.	RENTISTICO	CONCEPTO		TOTAL CONCEPTO
1	50005-01-03 CAMBIO DE MODALIDAD Y MODIFICA	12 MARCAS Y LEMAS COMERCIALES		111,000.00
		TOTAL :	$	111,000.00

SON: CIENTO ONCE MIL PESOS

RESPONSABLE : _____

RECIBO DE CAJA APLICADO AL EXPEDIENTE No. _____

REIVINDICACIONES

1. Método para la preparación de una composición que proporciona fragancia sólida de flujo libre, caracterizado porque comprende la adición de una fragancia en la forma de un líquido o una solución de solvente orgánico a un material portador particulado en presencia de una sal soluble en agua de un metal álcali o alcalino térreo, que comprende la mezcla uniforme de ingredientes secos (portador particulado, sal, cualquier otro ingrediente) seguido por la adición de la fragancia líquida y agitando hasta que se logra un polvo seco de flujo libre, el portador particulado es sílice porosa, fina, que contiene opcionalmente hasta 50% en peso de otros materiales particulados absorbentes, donde la cantidad de sal soluble en agua presente en la composición está entre 60% y 85% en peso y la cantidad de portador poroso se encuentra entre 5% y 20% en peso de la composición y la relación de sal soluble en agua a fragancia es desde 20:1 hasta 1.5:1.

2. Método de conformidad con la reivindicación 1, caracterizada porque la relación en peso del portador particulado a la sal soluble en agua es desde 1:1 hasta 1:20, más preferiblemente desde 1:5 hasta 1:20, aún más preferiblemente desde 1:10 hasta 1:20 y más preferiblemente desde 1:8 hasta 1:15.

3. Método de conformidad con la reivindicación 1 ó 2, caracterizado porque la relación en peso de la sal soluble en agua a perfume es desde 20:1 hasta 1.5:1, más preferiblemente desde 8:1 hasta 5:1 o desde 10:1 hasta 20:1.

4. Una composición que proporciona fragancia sólida de flujo libre, caracterizada porque consiste esencialmente de un portador particulado en el cual es depositado una fragancia y una sal soluble en agua de un metal álcali o alcalino térreo, la composición comprende entre 60% y 85% en peso de sal soluble en agua y entre 5% y 20% en peso de un portador particulado, el portador particulado en sílice porosa fina que opcionalmente contiene hasta 50% por peso de otros materiales particulados absorbentes y la relación de sal soluble en agua a fragancia es desde 20:1 hasta 1.5:1.

5. Composición de conformidad con la reivindicación 4, caracterizada porque la relación en peso del portador particulado a sal soluble en agua es desde 1:2 hasta 1:20, más preferiblemente desde 1:5 hasta 1:20, aún más preferiblemente desde 1:10 hasta 1:20 y más preferiblemente desde 1:8 hasta 1:15, y la relación en peso de sal soluble en agua a perfume es desde 8:1 hasta 5:1.

6. Composición de conformidad con la reivindicación 5, caracterizada porque la relación en peso del portador particulado a sal soluble en agua es desde 1:2 hasta 1:20, más preferiblemente desde 1:5 hasta 1:20, aún más preferiblemente desde 1:10 hasta 1:20 y más preferiblemente desde 1:8 hasta 1:15, y la relación en peso de sal soluble en agua a perfume es desde 10:1 hasta 20:1.

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(54) Method for preparing fragrance products

(57) The present invention relates to a process for reducing fragrance loss in a substrate. This process includes preparing a protected fragrance matrix that contains a perfume, an optional fixative and a solid absorbent. The process further includes combining a substrate conditioner with the fragrance matrix and a substrate. Products, especially fabric softener sheets, made from these processes are also provided.

EP 1 061 124 A1

Description

[0001] The present invention relates to substrates having prolonged fragrance. More particularly, the present invention provides compositions and methods for reducing the loss of perfume during manufacture.

[0002] Fabric softener sheets typically include a substrate, a fabric conditioner base and a fragrance. The fabric conditioner base is used to impart a benefit to a fabric treated therewith. Such a benefit may include, for example, softness, reduced static of dried articles, easier ironing, etc. The fragrance is a used to impart a pleasant aroma to the dried fabric.

[0003] In use, these fabric softener sheets are typically placed in a conventional dryer together with fabric to be dried, e.g., wet laundry. During the drying process, fragrance and other benefits are transferred from the fabric softener sheet to the fabric being dried.

[0004] Conventionally, a fabric softener sheet is manufactured by coating a mixture of fabric conditioner base and fragrance onto a substrate. Typically, fabric softener bases are solid mixtures that must be melted prior to use. In practice, fabric conditioner bases are melted in a holding tank at about 70°C to 90°C then fragrance is added. Depending on the speed of coating, the conditioner base and fragrance may remain at these elevated temperatures for 4 to 8 hours. Fabric softener sheets are coated by continuously passing a substrate, e.g., a polyester sheet material, at high speed through a coating tank that is also held at 70°C to 90°C. The coated sheet material is then passed through a series of cooled rollers and a cooling tower to reduce the sheet temperature to about 30°C.

[0005] Fragrance losses occur at various points during the conventional fabric sheet manufacturing process; these losses in total may be up to 30 percent to 45 percent.

[0006] Encapsulates, such as cyclodextrin compositions as outlined in U.S. Patent No. 5,348,667, have been used to reduce perfume loss during the manufacturing process. Such encapsulation technology, however, is expensive to make and use, and is therefore not cost effective in a large-scale commercial setting.

[0007] Absorbents have been used to carry lipophilic materials such as fragrance in the manufacture of certain products. For example, it is known that solids can adsorb oily materials, such as, perfume. Such adsorbent solids have been reportedly used as carpet cleaning powders (U.S. Patent No. 5,783,543 and U.S. Patent No. 5,286,400), in oil adsorbent products (U.S. Patent No. 4,537,877 and U.S. Patent No. 5,763,083), in detergent powders (U.S. Patent No. 5,656,584), in detergent liquids (U.S. Patent No. 4,983,422 and U.S. Patent No. 4,209,417), in detergent bars (EP 816484), and in liquid softeners (U.S. Patent No. 4,954,285).

[0008] Accordingly, one object of the present invention is to provide a process for producing a fabric softener sheet which process significantly reduces the loss of the perfume in the fragrance matrix during the manufacture of such a sheet compared to conventional processes.

[0009] Another object of the invention is to provide a substrate having prolonged fragrance compared to conventionally prepared substrates.

[0010] Another object of the invention is to provide an efficient, cost-effective method for reducing the loss of perfume during the manufacture of fabric softener sheets.

[0011] The present invention is directed to meeting these and other objects.

[0012] The present invention provides a process for reducing fragrance loss during the manufacturing of fragranced substrates. This process includes providing a protected fragrance matrix that contains a perfume, a solid absorbent and optionally a fixative. In this process, a fabric conditioner base is further provided. A substrate is then combined with the fragrance matrix and the fabric conditioner base. A substrate treated in this manner retains more of the fragrance compared to conventionally prepared substrates.

[0013] The present invention also provides a substrate impregnated with a fragrance matrix having prolonged organoleptic activity. This substrate is made by the process of forming a protected fragrance matrix that includes a perfume, an optional fixative and a solid absorbent. The fragrance matrix is then combined with a fabric conditioner base. The fragrance matrix and fabric conditioner base are then applied to the substrate either together as a mixture or sequentially. In this process, the fragrance matrix is combined with the fabric conditioner base immediately prior to application of the mixture to the substrate. Alternatively, the fragrance matrix is applied to the substrate immediately after the fabric conditioner base.

[0014] The present invention provides a low cost method of reducing the loss of perfume during the manufacture of, e.g., a fabric softener sheet. This process includes the addition of a protected, i.e., an enrobed perfume contained within a fragrance matrix to a fabric conditioner base prior to application of the fabric conditioner base onto or into a substrate or during one of the rolling stages of a conventional fabric softener sheet manufacturing process. By using the process of the present invention, it is easier to dose, i.e., apply the fragrance to the substrate during the manufacturing process when the perfume is in a solid carrier form.

[0015] Thus, the process of the present invention includes (1) preparing a fragrance matrix including an enrobed, i.e., a protected perfume, an optional fixative and a solid absorbent; and (2) combining the fragrance matrix with a fabric conditioner base and a substrate.

[0016] As used herein, the phrase, "fabric softener sheet" includes a substrate, a fabric softener base and a fragrance matrix. For purposes of the present invention, the term "substrate" is intended to mean any material that acts as a delivery vehicle for the fragrance matrix and fabric conditioner base. Thus, in the present invention, the substrate must be able to retain in its interior or on its surface a sufficient quantity of the fabric conditioner base and fragrance matrix required to deliver the desired performance (prolonged fragrance) to the fabric, e.g., a fabric softener sheet. Typically, the substrate is in the form of a sheet, although other forms may be used. The substrate may be a synthetic or natural fiber that is woven, knitted or extruded. Non-limiting examples of materials that may be used as a substrate in the present invention include cotton, Rayon, polyester, regenerated cellulose, and the like.

[0017] As used herein, the phrases "fragrance matrix" or "protected fragrance matrix" mean that at least the most volatile parts of a perfume are protected or enrobed by a solid absorbent and optionally a fixative to reduce the amount of loss of fragrance during the manufacturing process of a substrate through evaporation of the perfume volatiles.

[0018] In the present invention, the protected fragrance matrix is said to "enrobe" the perfume. Thus, as used herein, the terms "enrobe," "enrobed" or "enrobement" refer to the sequestration of the perfume in the protected fragrance matrix to reduce the loss of fragrance during the manufacture of fragranced substrates, e.g., fabric softener sheets, and to prolong the effect of a fragrance on substrates coated with such an enrobed perfume.

[0019] The protected fragrance matrix is preferably in the form of a flowable powder. Thus, the ratio of perfume, solid absorbents and fixative are carefully controlled to maintain the consistency of a flowable powder. In this embodiment, the perfume-to-solid absorbent ratio is between about 1:20 to about 2:5, preferably about 1:5. The level of perfume, however, will depend upon the final composition of the substrate. The fragrance, in turn, may be present in the protected fragrance matrix in an amount of from about 10% to about 50% (wt/wt), preferably, in an amount of from or between about 20% to about 40% (wt/wt). On the substrate, the fragrance level will be about 1% to about 6% (wt/wt).

[0020] The flowable powder may be dosed into, for example, a molten fabric conditioner base such as a softener or an antistatic system. Preferably, the powder is mixed into the fabric conditioner base immediately prior to application of the mixture to the substrate. Alternatively, the powder is applied to a substrate previously coated with the fabric conditioner base at a subsequent processing step, such as at one of the rolling stations during the manufacture of a fabric softener sheet.

[0021] As used herein, the term "immediately" is intended to mean that the fragrance matrix is added to the fabric conditioner base just prior to application to the substrate. Typically, the fragrance matrix is added 1-20 minutes prior to application to the substrate, preferably 2-10 minutes prior to application to the substrate; most preferably the fragrance matrix is dosed at the rolling stage directly onto the substrate coated with the fabric conditioner base.

[0022] In the present invention, the protected fragrance matrix may contain all or part of the perfume. If part of the perfume is protected, i.e., enrobed, then it is usual to select the most volatile components to be enrobed.

[0023] For example, in the present invention, the protected fragrance matrix is formed into a powder by mixing the perfume with an absorbent solid and an optional fixative. Absorbent solids that may be used in accordance with the present invention include, for example, clays, silicas, Celites® (ICN Biomedicals, Inc., Aurora, OH, USA.), zeolites, metal salts, including for example phosphates, cellulose, such as methyl cellulose, starches, carbonates, such as sodium bicarbonate, borates, such as sodium borate, sulfates, such as sodium sulfate, water-soluble polymers, borax and mixtures thereof.

[0024] For purposes of the present invention, the term "perfume" is intended to mean a mixture of a single or complex mixture of aromatic chemicals which have been formulated to give an aesthetically pleasing smell to a substrate, such as for example, a fabric softener sheet. As used herein, "aroma chemicals" are intended to mean chemicals that have an odor. There are many chemical classes which fall within the meaning of the phrase "aroma chemicals" as used herein, including for example, hydrocarbons, alcohols, aldehydes, ketones, lactones, esters. These chemical classes are described in S. Arctander Perfume Flavors and Chemicals, Vols. 1 and 2, Arctander, NJ, USA.

[0025] The perfume may also contain small amounts of other additives, such as solvents, preservatives, antioxidants, UV screening agents and the like. The fragrance matrix may also include organoleptic components, such as for example, other well-known fragrance ingredients.

[0026] Preferably, a fixative is incorporated into the fragrance matrix of the present invention. The fixative preferably is a high molecular weight, low melting solid composition that may be mixed into a powder. Examples of suitable fixatives for use in the present invention include polyethylene glycol, Glycerox (Croda Inc., Parsippany, NJ, USA.), mineral oil and mixtures thereof. In accordance with the present invention, the molecular weight of the fixative may vary between about 400 to 20,000 daltons, preferably between about 2,000 to 10,000 daltons. Other fixatives having a similar viscosity and melting point to polyethylene glycol are also contemplated by the present invention. The level of fixative useful in accordance with the present invention is between about 1% and about 40% (wt).

[0027] The solids and the liquids set forth above are combined by, for example mixing in a suitable mixer until the blend is flowable so that it is easy to apply (i.e., dose) to a substrate.

[0028] The present invention also includes a fabric conditioner base. For purposes of the present invention, the fabric conditioner base includes a mixture of ingredients that impart a desired property to the fabric, such as for example,

softness, no static cling, reduced creasing, easier ironing, and the like. These properties are achieved using, for example, cationic, zwitterionic, and non-ionic softeners, soaps and quaternary compounds, as well as other fatty materials alone or in combination. Other examples of suitable fabric softener bases may be found in U.S. Patent No. 3,686,025.

[0029] Other optional components may also be added to the fabric conditioner base to further impart various desired characteristics to the substrate, so long as these ingredients do not significantly interfere with the reduction of fragrance loss during manufacturing of substrates. Such optional components include, for example, antistatic agent(s), anti dye-transfer agent(s), soil release agent(s), and the like.

[0030] In the process according to the present invention, the substrate may be coated at several carefully timed points and according to a specific sequence of events to achieve the desired fragrance effect. For example, in one embodiment, the substrate may be coated by combining the fragrance matrix with the fabric conditioner base to form a mixture immediately prior to applying the mixture to the substrate. Alternatively, the substrate is coated by applying the fabric conditioner base thereto to form a substrate-fabric conditioner base composition. Thereafter, the fragrance matrix is applied to the coated substrate.

[0031] For purposes of the present invention, the fragrance matrix and/or fabric conditioner base is applied to the substrate using any conventional technique suited for the particular application, such as, for example, spraying, coating, dipping and the like. Using these and other techniques, the fragrance matrix and fabric conditioner base are applied to the substrate as a coating. Alternatively, the fragrance matrix and fabric conditioner are impregnated throughout the substrate, i.e., are dispersed within the interstitial matrix of the substrate as well as its surface.

[0032] Preferably, the fabric conditioner base and fragrance matrix are dispersed throughout the substrate to a uniform thickness of about 0.1 to about 2 mm to evenly distribute the fragrance throughout the entire substrate.

[0033] Another embodiment of the present invention is a substrate impregnated with a fragrance matrix having prolonged organoleptic activity. This substrate is made by the process which includes (a) forming a protected fragrance matrix as set forth previously with a perfume, a fixative and a solid absorbent; and (b) mixing the fragrance matrix with the fabric conditioner base immediately prior to applying the mixture to the substrate or applying the fragrance matrix to the substrate immediately after the fabric conditioner base is applied to the substrate.

[0034] Thus, in one embodiment, a mixture of the fragrance matrix and fabric conditioner base is applied to the substrate. In this embodiment, the fragrance matrix is added to the fabric conditioner immediately prior to application of the mixture to the substrate. Alternatively, the fabric conditioner base and fragrance matrix are applied to the substrate sequentially. In this embodiment, the fabric conditioner is applied to the substrate first, immediately followed by the fragrance matrix.

[0035] The fixative according to this embodiment is selected from high molecular weight, low melting point compositions such as for example, polyethylene glycol, Glycerox, mineral oil and mixtures thereof.

[0036] The following examples are set forth to illustrate the processes and compositions of the present invention. These examples are provided for purposes of illustration only and are not intended to be limiting in any sense.

EXAMPLES

Example 1

Enrobed Composition

[0037] An enrobed, i.e., protected fragrance matrix was made by combining the ingredients as set forth below:

Component	%
Perfume Oil (Givaudan Roure) Fragrance PNF217FPN	20
Polyethylene Glycol 4000 (Union Carbide)	1
Bentonite H (Whitaker, Clark & Daniels)	10
Valfor® 100 Aluminosilicate Zeolite (PQ Corp.)	69

Example 2

Comparison of Fragrance Retention of Enrobed Composition vs. Control

[0038] A comparison between a fabric softener sheet made using a conventional process and a fabric softener sheet made according to the processes of the present invention are set forth below.

Control

[0039] A conventional fabric softener base, Varisoft 136-100 (Witco, Inc. OH) was heated to 80° C in a convection oven. 48 grams of this composition was placed in a pre-heated beaker and 2 grams of the perfume oil PNF217FPN (fragrance level = 4%) was added to the beaker with agitation. The mixture was held at 80° C for 4 hours to simulate production and then applied to a commercially available polyester softener sheet in a pilot plant using a coating machine (Talbot Engineering Corp.).

Enrobed Composition

[0040] Varisoft 136-100 (Wites Corp., Dublin, OH, USA) was heated to 80° C and 40 grains thereof was placed in a pre-heated beaker as described above. 10 grams of the protected fragrance matrix (20% PNF217FPN = 4% fragrance) according to Example 1 was added to the Varisoft with mixing immediately prior to application of the mixture onto a fabric softener sheet in a pilot plant.

[0041] On cooling, the fragrance level in the control and enrobed softener sheets was determined by extraction and analysis by gas chromatography/mass spectrometry. The results are shown below:

Percent of theoretical fragrance level	
Control Sheet	28.8
Enrobed Sheet	43.2

[0042] As the data above indicates, perfume loss in the pilot plant was reduced from 71% to 57% using the late addition of a protected fragrance matrix (Enrobed Sheet) compared to the control sheet.

Example 3

Enrobed vs. Unprotected Compositions in Open/Closed Systems

[0043] The manufacture of fabric softener sheets requires that a melted fabric softener base and a fragrance be held in an open/closed vessel at 80°C (176°F) for up to 4 hours. To demonstrate the ability of the present enrobed processes to reduce fragrance loss, two samples were set up as follows:

Control (Non Protected Perfume)

[0044] 48 grams of Varisoft 136-100 was melted in a convection-draft oven at 80°C (176°F). 2 grams of PNF217FPN (Fragrance level = 4%) was added to the Varisoft composition and held at 80°C for 4 hours in an open beaker.

Enrobed Composition

[0045] 40 grams of Varisoft 136-100 base was melted in a convection-draft oven at 80°C (176°F) and 10 grams of a protected fragrance matrix according to the present invention (20% PNF217FPN = 4% fragrance level) from Example 1 was added to the base. The mixture was held at 80°C for 4 hours in an open beaker.

[0046] Both samples were extracted and analyzed. The results are shown in Table 2.

United States Patent [19]

King

[11] Patent Number: 5,041,421

[45] Date of Patent: Aug. 20, 1991

[54] FRAGRANT MATERIAL

[75] Inventor: Michael L. King, Decatur, Ill.

[73] Assignee: Mari-Mann Herb Co., Inc., Decatur, Ill.

[21] Appl. No.: 428,176

[22] Filed: Oct. 27, 1989

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 266,792, Nov. 3, 1988, abandoned.

[51] Int. Cl.[5] A61K 7/46

[52] U.S. Cl. 512/4; 424/76.3; 424/76.4; 424/84

[58] Field of Search 512/1, 2, 5; 424/76.3, 424/76.4, 84; 426/4

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Sale of foam plastic pearls.

Primary Examiner—James H. Reamer
Attorney, Agent, or Firm—Fish & Richardson

[57] ABSTRACT

A fragrant material that imparts a pleasant fragrance when set out at ambient temperature or when placed in heated water. The fragrant material comprises an aggregate of small sodium chloride granules such as are produced by vacuum granulation, the granules formed into a molded shape, e.g. as a plurality of discrete pellets, or in a loose state, and having a fragrant oil and optionally a water soluble dye dispersed in the material. Also disclosed is a fragrant material comprising the combination of salt granules with a minor amount of finely divided inert particles, and a water-soluble dye. Other features that enhance performance and enable the product to be of low cost are also described.

39 Claims, No Drawings

FRAGRANT MATERIAL

CROSS REFERENCE

This application is a continuation in part of U.S. application Ser. No. 07/266,792 filed Nov. 3, 1988, by Michael L. King and entitled "Fragrant Material", now abandoned.

FIELD OF THE INVENTION

This invention relates to fragrant material for imparting a pleasant fragrance to a room, and the like.

BACKGROUND OF THE INVENTION

Man has imparted pleasant fragrances to rooms since time immemorial. Flowers, eucalyptus leaves, dill, and incense have been used for this purpose for thousands of years and are still widely used today. A wide variety of fragrant oils are now commercially available, but they are rarely used by themselves for providing fragrance to rooms because of their rapid evaporation rate and because of the danger of spills. Instead, fragrant oils are generally incorporated into a solid carrier, also known as a fixative. A number of different prior art carriers are or have been used with fragrant oils. For example, Moore, U.S. Pat. No. 4,427,366, discloses wax chips containing a fragrant oil and Gyulay, U.S. Pat. No. 4,579,717, discloses a porous ceramic ring which absorbs a fragrant oil. Another well known carrier is a mixture of dried Orris root, dried Calamus root, and other dried plant material which, when scented with a fragrant oil, is commonly sold as potpourri Rock salt to which fragrance oils have been applied has also been sold. However, none of these carriers is ideal.

An ideal carrier for a fragrant oil, depending upon the application, would exhibit many specific properties. First, the carrier would readily absorb fragrant oils and would leave no surface film which might cause staining or tackiness. Second, the carrier would also readily absorb dyes to permit the carrier to be dyed a color corresponding to the fragrance if desired. Third, the carrier would be odorless so as to not clash with the fragrance of the oil. Fourth, it would be nonflammable, nontoxic, nonallergenic, non nutritive and environmentally safe for obvious safety reasons. Fifth, for particular applications, an ideal carrier would consist of discrete particles having an average size of about ¼ to 1 inch with little or no fines. Such particles can be easily mixed, poured, and handled; they attractively fill up bowls and containers of various sizes and shapes; and, if spilled, can be picked up by hand. Sixth, the carrier would not soften or melt at elevated temperatures of 100° to 130° F. which are encountered in closed houses and automobiles during summer months. Seventh, the carrier would be relatively inexpensive and readily available.

Furthermore, an ideal carrier would be suitable for "simmering"—warming the material in or out of water to accelerate the flow of fragrance into the room. More particularly, the carrier would dissolve slowly in water, cause no harm to pots and pans and permit an easy clean up after use.

A large number of potential carriers were considered and/or tested and then rejected before the fragrant material of this invention was discovered. For example, potpourri crumbles into fines and leaves a messy residue after simmering in hot water. Wood chips exhibit similar disadvantages and have too strong an odor. Sugar cubes readily absorb oils and dyes, but become sticky and attract bugs. Starch readily absorbs oils and dyes, but cannot be easily formed into lasting solid shapes, attracts bugs and leaves a messy residue when used with water. Rock salt does not absorb oil and dye well because of its structure. Wax chips must be melted to incorporate the fragrant oil and dye, release little fragrance until they are heated, and create a mess if placed directly into hot water. Porous ceramic materials have limited absorbency, are expensive, and present a danger if broken because of the sharp and jagged edges of the pieces. Cellulose fibers are dry and absorb oils and dyes, but are not water soluble and are expensive. Vermiculite is also dry and will absorb oils, but will not dye well, is not water soluble, and is expensive. Accordingly, until the fragrant material of this invention was discovered, there existed a strong demand for a better solid carrier for fragrant oils.

SUMMARY OF THE INVENTION

The object of this invention is to provide an improved fragrant material for imparting a pleasant fragrance to a room or the like. A more particular object is to provide such a fragrant material which can be set out at ambient temperature in a room or placed in heated water.

I have discovered a fragrant material which meets these objects. According to one aspect of the invention, the material comprises a plurality of discrete pellets of compressed salt having a fragrant oil dispersed within the pellets. This fragrant material exhibits many advantages over other types of fragrant material which have been previously used in homes. The material has no surface film so it is non-staining. The material is odorless, nonflammable, nontoxic, nonallergenic, non nutritive, environmentally safe, homogeneous, non-crumbling with a high pressure break strength, and does not melt even at elevated temperatures. The material is relatively inexpensive and can be easily mixed with other fragrant materials. Furthermore, this material dissolves slowly in water, does not harm pots and pans, and can be easily cleaned after use. Finally, because the fragrant oil, and dye if present, is uniformly dispersed throughout the pellets, a partial dissolution of the pellets does not remove all the fragrant oil and dye from the pellets.

In another aspect, the invention features a fragrant material for imparting a pleasant fragrance when exposed in dry state at ambient conditions or when placed in heated water, that includes a molded shape of compressed sodium chloride salt granules having a fragrant oil dispersed within the molded shape.

Preferred embodiments of this aspect of the invention include additional features. A plurality of the molded shapes in the form of pellets are provided having an average size of approximately ¼ to 1 inch. The granules of the molded shape comprise vacuum granulated sodium chloride. The fragrant material additionally includes a dye dispersed within the molded shape. The fragrant material is formed by the process of applying the fragrant oil to the surface of a pre formed shape of the compressed salt granules and allowing the oil to be absorbed into the pores between the granules; preferably the oil is applied by spraying. The fragrant material is formed by the process of applying the fragrant oil to a loose aggregation of granules of sodium chloride and thereafter compressing the granules into the molded

shape; preferably high molding pressure is employed to form the molded shape.

In another aspect, the invention features a fragrant material for imparting a pleasant fragrance when exposed in dry state at ambient conditions or when placed in heated water, that includes an aggregation of small vacuum granulated sodium chloride granules and a fragrant oil dispersed within the aggregation. In addition to including embodiments in which the aggregation of granules has been formed into a molded shape, this aspect of the invention also includes embodiments in which the small salt granules remain loose as a powder.

In embodiments of the various aspects of the invention described above, certain additional features are preferred. Most of the sodium chloride granules are about 850 microns or less in size. The aggregation includes a minor amount of finely divided, inert, water insoluble, hygroscopic particles that increase the carrying capacity of the mixture for the fragrant oil; preferably these particles are selected from the group consisting of silicon dioxide, calcium silicate, magnesium carbonate, tricalcium phosphate and sodium silicoaluminate and mixtures and combinations thereof; preferably they have a particle size of about 10 microns; preferably the weight ratio of the finely divided particles to the fragrant oil is about 1 to 3; preferably the finely divided particles comprise about 2% or more by weight of the fragrant material and more preferably to enhance a fragrancing effect as described while still being economical, the finely divided inert particles comprise more than about 3% but less than about 10% by weight of the fragrant material. The fragrant oil of the fragrant material includes an organic carrier fluid selected from diethylphthalate, methyl dihydroabietate, Hercolyn D, propylene glycol, dipropylene glycol, isoparaffins, Losol, saltrol, polysorbate, D limonene, glycerine and Isopar M; preferably the fragrant oil has a viscosity under ambient conditions of about 10 centipoise or more, preferably on the order of between about 10 and 20 centipoise. The fragrant material further includes a dye which is soluble in water, and perferably also in oil; the dye preferably is a polymer-bound azo colorant.

In another aspect, the invention features a dry, loose mixture comprising at least an aggregation of a large number of small vacuum granulated sodium chloride granules, a relatively minor amount of finely divided, inert, water insoluble, hygroscopic particles and a fragrant oil dispersed within the aggregation.

Another aspect of the invention is a fragrant material for imparting a pleasant fragrance when exposed in dry state at ambient conditions or when placed in heated water the fragrant material comprising an aggregation of small anhydrous salt granules and a minor amount of finely divided, inert, water soluble, hygroscopic particles, the finely divided inert particles selected from the group consisting of silicon dioxide, calcium silicate, magnesium carbonate, tricalcium phosphate and sodium silicoaluminate and mixtures and combinations thereof, the aggregation carrying a fragrant oil, the weight ratio of the finely divided particles to the fragrant oil being about 1 to 3, the salt granules in the fragrant material being selected so that in the presence of the inert particle they are dimensionally stable under ambient conditions.

Preferred embodiments also include selection of the ingredients to preform various functions The fragrant oil is selected to perform as a room freshener. The fragrant oil is selected to mask a pervading odor such as, for example, an animal odor. The fragrant material is adhered as a coating to an object to which it is desired to impart a fragrance. A dry mixture may be, for example, formulated to mask a pervading odor, e.g., in the form of a rug freshener.

The invention also includes the methods of forming the various materials described above.

DETAILED DESCRIPTION OF THE INVENTION

In certain important embodiments of the invention the fragrant material of this invention comprises discrete pellets of compressed salt, fragrant oil, and, optionally, dye. If the fragrant material is going to be simmered in water, it is preferred that the salt contain minimum amounts of insolubles. If the fragrant material is not going to be simmered, the purity of the salt is not critical. The preferred type of salt is sodium chloride (NaCl) because of its low cost and ready availability. The physical form of the pellet is compressed granular salt. This form has a high pressure break strength and yet is still extremely absorbent because of the microscopic pores between the compressed salt granules. For certain applications, the salt pellets have an average size of approximately ¼ to 1 inch, or about the size of marbles. For many applications, pellets of a smaller size tend to dissolve too quickly in water and are difficult to pick up if spilled, while pellets of a larger size dissolve too slowly in water and are not as attractive when placed in small bowls and containers. The shape of the pellet is not critical and spheres, cubes, cylinders, and the like are acceptable. There is no requirement that the shape and size of the pellets be uniform and, in fact, some variation is aesthetically pleasing. One preferred form of salt pellet is of the type used commercially to recharge water softeners. A representative example of such a pellet is the Morton ® System Saver TM water softening pellet. These pellets typically contain about 99.70 percent sodium chloride and are about 1 inch long, ⅝ inch wide, and ⅝ inch thick.

A fragrant oil is a concentrated liquid fragrance. The fragrant oil may be derived from natural sources or may be synthetic. The oil generally contains a variety of organic compounds of differing volatilities. The particular type of fragrant oil is not critical to this invention and many suitable fragrant oils are commercially available. A more complete discussion of fragrant oils is found at Volume 14,page 717, et seq. under the heading "Perfumes" in the Kirk-Othmer Encyclopedia of Chemical Technology (2d Ed. 1967).

The optional dye is a substance which imparts color to the fragrant material. The dye may be available as a powder or as a liquid. Dyes which are water soluble are generally preferred because they have less tendency to stain and can be cleaned more easily. The particular dye used in this invention is a matter of choice and many suitable dyes having good color stability and retention are commercially available.

The relative amounts of salt pellets and fragrant oil in the material is a matter of choice which depends upon the strength of the fragrance in the oil and the desired strength of the fragrance in the fragrant material. The weight ratio of salt pellets to fragrant oil is generally about 20:1 to 100:1, and preferably about 30:1 to 50:1. If excessive amounts of fragrant oil are used, the saturation point of the salt pellets may be exceeded and the pellets may have an undesirable "wet" surface. If a dye is used, its quantity is also a matter of choice.

It is found while achieving a dry-to-the-touch character it is possible to include larger amounts of fragrant oil in the material by addition of the finely divided inert additive mentioned above in quantities greater than what might ordinarily be included as free flowing agents in common salt. (In the prior art such agents have been used for the purpose of making salt free flowing or capable of being compressed into the form of a pellet.) The quantity of the particles is generally about 2% or more; in many cases, especially for free flowing powder embodiments, in the range of about 3 to 10% of the total weight of the fragrant material is employed.

Further details concerning preferred ingredients and uses will be given following the description of certain examples.

EXAMPLE 1

An 80 pound plastic film bag of Morton ® Salt Pellets was opened and the contents gently poured into an enamel cast-iron 60 gallon tub. The salt pellets are a commercial product of the Morton Salt Division of Morton Thiokol, Inc., Chicago, Ill. They are compressed from vacuum granulated salt and contain a minimum of 99.5 percent sodium chloride. The dimensions of a typical pellet are 1 inch long, ⅝ inch wide, and 3.8 inch thick.

Two pounds of peach fragrant oil, a commercial product of The Lebermuth Company, South Bend, Ind., are poured into a standard 2.5 gallon hand sprayer. Two ounces of Liquidtint Yello BL dye, a commercial product of the Millikin Dye Company, Inman, S.C., are then poured into the sprayer. The sprayer is then sealed and vigorously shaken to uniformly mix the fragrant oil and dye.

The sprayer is then pumped to generate pressure and the contents are sprayed upon the pellets. The pellets are mixed during spraying to obtain uniform distribution. After spraying, the pellets are allowed to sit for about one hour to allow complete absorption of the oil and dye. The fragrant material is then screened to remove fines and packaged.

EXAMPLE 2

This example is formed by application of a fragrant oil to salt granules prior to compression of the granules to form a pellet. A vanilla-creme fragrance containing vanilla-creme fragrant oil, Ethyl Vanillin, and Coumarin is obtained. (The fragrance supplier may include small quantities of other ingredients according to its recipe). The fragrance components are added to approximately 30% carrier fluid (Diethyl Phthalate) forming a fragrant oil with the following properties:

Specific Gravity @20° C. (water = 1)	1.1328–1.1338
Refractive Index @25° C.	1.5155–1.5165
Vapor Density (AIR = 1)	>1
Boiling Point (of most volatile component >1%)	over 175° C.
Solubility in water	neglible
Appearance and odor	colorless to pale yellow liquid with a strong vanilla fragrance.

The fragrant oil is then added to an anhydrous dendretic sodium chloride salt including about 2 percent of Cabosil. About 2.5% of fragrant oil by weight is added to the salt (about 50 lbs. oil per 2000 lbs. of salt). The fragrant oil is mixed with the salt and Cabosil mixture to form a slurry. The slurry is gravity fed into a tablet press that exerts close to 1500 lb./sq. in. of pressure, achieving temperatures close to 806° C. (near the melting point of NaCl granules at the high pressure), which results in fusion of the granules, with the formation of "bridging" areas where the granules are bonded together. After tabletizing the final pellets are extremely hard, shiny, solid and have a glazed appearance and are stable, non-crumbling, and smell like vanilla.

In use, with respect to the foregoing examples, there is no need of inducing an airflow because the fragrance is sufficiently strong to disperse on its own. The pellet or tablets will "fragrance" an average sized open room of 100 sq. ft. for at least 6 months, and are also highly water soluble.

A by product of producing the pellets or tablets is the extra powder or granules that are vibrated off through a screen. This powder has all the properties of the pellets or tablets except that it is in powder form. It is found to have a number of practical applications, for instance, for placing in heated water, or as a fragrance powder, e.g. to mask a pervading odor such as the smell of urine.

EXAMPLE 3

A fragrant material is formed by application of a fragrant oil to salt granules prior to compression of the granules to form a pellet A cinnamon fragrance containing cinnamon fragrant oil, Cinnamine Aldehyde, Cinnamon Leaf Oil and Ethyl Vanillin is obtained. The fragrance components are added to approximately 20% carrier fluid (7% Hercolyn-D, 13% dipropylene glycol), forming a fragrant oil with the following properties:

Specific Gravity @20° C. (water = 1)	1.048–1.058
Refractive Index @25° C.	1.581–1.591
Vapor Density (AIR = 1)	1
Boiling Point (of most volatile component 1%)	over 175° C.
Solubility in water	Neglible
Appearance and odor	pale yellow liquid with a cinnamon fragrance.

The fragrant oil is mixed with dye. By volume, 3 cc yellow Chromatech dye and 9 cc red Chromatech dye per 1 lb. of oil are used. The dyed fragrant oil is added to dendritic sodium chloride salt and about 2 percent Cabosil. About 2.5% of fragrant oil by weight is added to the salt (50 lb. oil/2000 lb. of salt). The oil is mixed with the salt to form a slurry and processed as in Example 2.

EXAMPLE 4

In this example, a loose form (powder) fragrant material is formed by application of a fragrant oil to a mixture of salt granules and particles that enhance carrying capacity. The material is formed as powder by addition of a fragrant oil to a non-compressed mixture of salt granules and a small amount of the finely divided particles. The amount of the enhancing particles is used in excess of 3% and generally less than about 10% by weight of the amount of salt, in this example, 5%.

A wintergreen fragrance containing wintergreen fragrant oil, peppermint, spearmint and wintergreen oil is obtained. The fragrance components are added to approximately 40% carrier fluid (isoparaffins), forming a fragrant oil with the following properties:

US005840668A

United States Patent [19]

Behan et al.

[11] Patent Number: 5,840,668

[45] Date of Patent: *Nov. 24, 1998

[54] PERFUMED LAUNDRY DETERGENT POWDERS

[75] Inventors: **John Martin Behan**, Kennington; **Richard Arthur Birch**, Hythe; **John Leake**, Chester; **Jeremy Nicholas Ness**, Canterbury; **Anthony Nock**, Warrington; **Keith Douglas Perring**, Ashford, all of Great Britain

[73] Assignee: **Quest International B.V.**, Naarden, Netherlands

[*] Notice: This patent issued on a continued prosecution application filed under 37 CFR 1.53(d), and is subject to the twenty year patent term provisions of 35 U.S.C. 154(a)(2).

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§ 102(e) Date: **Nov. 14, 1995**

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[52] **U.S. Cl.** **510/349**; 510/101; 510/102; 510/103; 510/104; 510/105; 510/106; 510/107; 510/349; 510/438; 510/441; 510/446; 510/466

[58] **Field of Search** 510/105-107, 510/349, 438, 441, 446, 466; 512/4

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[57] **ABSTRACT**

The invention concerns perfumed, free flowing concentrated laundry detergent powders (concentrates), having a ratio of total solid constituents to total liquid constituents of less than 30:1, wherein the perfume is sorbed onto carrier particles which release at least 60% w/w/ of the perfume in the Perfume Release Test and which are inert as defined by the Perfume Stability Test. The invention also concerns perfume-on-carrier systems designed for perfuming free flowing concentrated laundry detergent powders. Preferably the carrier particles are inorganic and have a perfume content of at least 25% w/w.

10 Claims, No Drawings

PERFUMED LAUNDRY DETERGENT POWDERS

FIELD OF THE INVENTION

The invention relates to perfumed laundry detergent powders and to perfume-on-carrier systems for incorporation into laundry detergent powders.

BACKGROUND OF THE INVENTION

Detergent powders for laundry use span a wide range of compositions. Traditional (or "regular") products are typified by a detergent surfactant level of between 8% and 20% by weight in total, more commonly 10% to 15%. The surfactants may be anionic, non-ionic, cationic, zwitterionic or amphoteric in nature, and commercial products may contain all classes of surfactants, but the predominant form is generally anionic (i.e. anionic surfactants typically account for 50% or more of the total surfactants). Typical detergent surfactants are described in detail in "Surfactant Surface Agents and Detergents", volume II by Schwartz, Perry and Birch, Interscience Publishers (1958). The remainder of a laundry detergent composition generally comprises builders, fillers, moisture, soil release and soil suspension and anti-redeposition agents, and other optional adjuncts such as processing aids, optical brighteners, dyes, foam control agents, anti-corrosion agents, perfumes, pH control agents, enzymes, stabilisers, bleaches and bleach activators. The level of solid components in regular laundry detergent compositions is high, usually above 75%, often above 85%. Perfume loadings for such compositions are generally within the range 0.05% to 0.4%, more commonly 0.1% to 0.3%, and the ratio of solid constituents to organic liquid constituents in a regular detergent composition is usually at least 30:1, and is likely in practice to be considerable higher, e.g. at least 150:1 and up to 500:1.

Laundry detergent powder concentrates and hyperconcentrates (for the purposes of this specification further referred to as "concentrates") represent a relatively new product segment which is assuming increasing commercial importance world-wide. These concentrated products have a rather different composition to that described above.

The total level of detergent surfactants in concentrates generally lies within the range 15% to 60% by weight of the powder, more usually 20% to 40%. In addition to the difference in the level of surfactants another major point of difference concerns the level of low-functionality materials such as fillers. In concentrates the level of sodium sulphate for example, is rarely above 6% or even 2% by weight, whereas in regular powders levels of 20% to 30% are common. The composition of the actives may be similar to that in regular products, i.e. predominantly anionics, but not restricted to this and, for example, a high proportion of non-ionics may be used advantageously. The use of higher proportions of non-ionic surfactants is reported to be a significant trend in the detergent industry, at least for Europe, as reported by Smulders and Krings (Chemistry and Industry, March 1990, pages 160 to 163). Examples of detergent powder formulations with high non-ionics levels are disclosed in EP 228011, EP 168102, EP 425277, and EP 120492. Many non-ionic surfactants are liquid at ambient temperatures. Yet another difference between "regulars" and concentrates is that the percentage of perfume incorporated into concentrates tends to be higher than that for regular powder use, and lies generally above 0.1% by weight, normally within the range 0.4% to 2.5% by weight of the powder, more typically 0.5% to 1.5%.

Thus a concentrate may be defined for the purposes of the present specification as comprising at least 15% (and preferably at least 20%) w/w total surfactant actives and at least 0.1% w/w perfume, preferably at least 0.4% w/w. A consequence of these differences between regulars and concentrates is that the ratio of total solid to total organic liquid components for certain concentrates may be less than 30:1, or even less than 10:1, and problems can arise in manufacture and storage of the product due to "stickiness" and lump formation. In particular, for concentrates incorporating relatively high levels of liquid non-ionic surfactants, processing problems may arise from agglomeration of particles, with handling difficulties during production, and with the possibility of forming large powder conglomerates which are unacceptable to the end user of the product. Even after optimisation of the powder formulation it may be found that the addition of further liquid components such as perfumes to these systems will exacerbate the tendency for particle cohesion and agglomeration, especially when the perfume is post-dosed into base powder, and when high levels of perfume are incorporated.

The prior art literature provides many instances where perfume is delivered via the use of a solid carrier, which may be either organic or inorganic in nature, or both.

GB 2066839 discloses detergent powders containing perfume sorbed onto a porous carrier at a level of 10% to 65% by weight. The carrier is a large surface area sorbent selected from a wide variety of substrates such as polyacrylates, polyvinyl chloride, urea-formaldehyde resins, polystyrene, hydrated silicon dioxide, titanium dioxide, aluminium trioxide, or mixtures thereof. The primary objective of the invention disclosed therein is to decrease perfume losses through volatilisation and chemical attack during storage of perfumed powder products.

GB 1306924 describes perfume particles in free-flowing powder form" prepared from perfume and a particulate silica, or more particularly from a mixture of a finely divided silica and an absorbent silica gel. The fragrance loading in these particles may be as high as 70%. Manufacture is economical and involves simple mixing of perfume oil with the silicas, followed by sieving. Examples are disclosed of perfume particles suitable for use in talcum powders, scouring powders, deodorants, toilet blocks, bath crystals and foot powders. No disclosure is made of use in detergent powders, and nothing is revealed regarding perfume stability within concentrates, nor are specific physical properties such as hydrophilic character or bulk density disclosed.

U.S. Pat. No. 4209417 teaches the use of perfume particles prepared using a water-soluble polymer together with an emulsifier. The loading of perfume in these particles may be high (30% to 70%), but the manufacturing process involves drying large volumes of aqueous dispersions, with adverse consequences for throughput and economy. In addition, the perfume must be water-insoluble. The teachings of GB 2090278 are similar, except that the perfumed particle contains water but is rendered free flowing by the inclusion of excess powdered hydratable material such as anhydrous alkali metal salts. The maximum possible loading of perfume is thereby significantly reduced.

Perfume-on-carrier systems for laundry detergent compositions as disclosed in EP 334666 are designed to deliver perfume to fabric efficiently through the use of fabric substantive carriers such as selected solid nonionic or cationic surfactants. The processing involves melt blending the components followed by spray chilling, and the resulting perfume loading is likely to be low (25% w/w or less).

Similar targeted delivery of perfume is provided by the microcapsules described in EP 376385, where a complex coacervate prepared from perfume and polycationic and polyanionic materials is coated with a water-insoluble cationic fabric softener which facilitates perfume delivery to fabric. In both these inventions the cost of manufacture is relatively high and relatively complicated processing steps are involved.

GB 2140820 and GB 2141730 describe perfume-on-carrier systems based on zeolites or clays, with an optional coating of a fabric-adhesive agent, wherein the perfume loading in these carriers is low (10% or less). The carrier systems are designed to deliver perfume to fabric via the zeolite or clay, which are likely to contact fabric and be dispersed thereupon during laundering, and are hydrophobic enough to retain sufficient perfume even in the wash solution.

EP 332259 and EP 332260 detail the use of silica perfume carriers in detergent powders and fabric softeners, respectively. The silicas span a wide range of types, with particle size of from 0.001 micron (fumed silica) through to 15 micron (silica gel), with a surface area of 100–800 m^2/g. For laundry detergent powders the preferred silica is a fumed type, with particle size in the range 0.007 micron. to 0.025 micron, although silica gels may also be used, the preferred particle size being 1 to 8 micron. These silica carriers are optimised for application in detergent compositions providing perfume at a level of 0.01 to 0.5%, and where the perfume includes components which require protection from hostile bleaching agents present in the detergent composition. The detergent compositions contain preferably 10% to 25% of detersive surfactants. The examples given are high in anionic type surfactants, and have high solid to organic liquid ratios. It is stated in EP 332259 that "in addition to separating and protecting the perfume from the bleach, it is believed that the silica particles deposit on fabrics and enhance the delivery of the perfume to the fabrics".

SUMMARY OF THE INVENTION

It is the purpose of the present invention to provide a method for incorporating perfumes in particulate form into concentrates so as to not adversely affect the flow properties of the detergent powder by the addition of perfume. It is another purpose of the invention to provide perfume-on-carrier systems for incorporation into concentrates in which perfumes retain their integrity and which can release substantially all their fragrance to the wash liquor during conventional laundering.

DETAILED DESCRIPTION OF THE INVENTION

In one aspect the invention provides perfumed, free flowing concentrated laundry detergent powders (concentrates), having a ratio of total solid constituents to total liquid constituents of less than 30:1 and containing at least 0.1 w.w of perfume, wherein the perfume is sorbed onto carrier particles which release at least 60% w/w of the perfume in the Perfume Release Test as described herein below and which are inert as defined by the Perfume Stability Test described herein below.

In another aspect the invention provides perfume-on-carrier systems wherein the carrier particles release at least 60% w/w of the perfume in the Perfume Release Test as described herein below and are inert as defined by the Perfume Stability Test described herein below.

For the purposes of this invention a concentrated laundry detergent powder (or "concentrate") is defined as a detergent powder comprising:

- at least 15% w/w, preferably at least 20% w/w, more preferably between 20 and 60% w/w of detergent surfactants (for brevity hereinafter referred to as "surfactants").
- less than 10% w/w, preferably less than 6% w/w, more preferably less than 2% of fillers, such as sodium sulphate.

Furthermore concentrates preferably have a bulk-density of at least 600 g/l.

Finally, concentrates generally contain other components, such as builders (phosphates such as sodium tripolyphosphate, zeolites or NTA), soil suspension and anti-redeposition agents, enzymes, optical brighteners, processing aids, dyes, foam control agents, anti-corrosion agents, pH control agents, stabilisers. Bleaching detergents also contain bleaches and bleach activators.

The invention is particularly concerned with concentrates in which a substantial proportion, i.e. 35% w/w or more, of the surfactants is liquid at ambient temperature. In this respect it should be taken into account that in many cases a particular surfactant is in fact a mixture of chemically closely related but slightly different components, e.g. a mixture of polymers with varying degrees of polymerization. Therefore, such products do not have a sharp melting point. For the purposes of this invention a surfactant is considered liquid when at a temperature of 25° C. and above it contains no more than 50% solids.

Under this definition the vast majority of anionic, cationic, zwitterionic and amphoteric surfactants are solids and surfactants falling in the category of liquids are predominantly nonionics, although many other nonionics are solids.

The invention is therefore also particularly concerned with concentrates in which a substantial part of the surfactant is of the nonionic type. In one embodiment it is concerned with concentrates comprising mainly nonionic and anionic surfactants in which the ratio of nonionic to anionic surfactant is at least 3:2 and wherein at least 35% w/w of the total quantity of surfactants is liquid. Thus, in these concentrates generally at least 60% w/w of the nonionics is liquid. In another embodiment it is concerned with concentrates in which all of the surfactants are nonionics, of which at least 35% w/w is liquid.

The concentrates according to the invention contain the perfume-on-carrier systems according to the invention in a quantity to give at least 0.1% w/w (calculated on the total concentrate) of perfume in the concentrate, preferably 0.4–2.5% of perfume, more preferably 0.5–1.5% w/w of perfume in the concentrate. The actual quantity of perfume-on-carrier system to be added thus also depends on the perfume content in the perfume-on-carrier system as hereinbelow described.

As used herein the term "perfume" denotes a substantially water-insoluble composition of matter consisting of one or more perfume components, optionally mixed with a suitable solvent or diluent, which is used to impart a desired odour to the detergent product to which it is added and/or to the wash liquor, the skin, hair or the fabric.

Perfume components are those constituents of a perfume which are added thereto only or primarily for their olfactive contribution. Perfume components may be natural products such as extracts, essential oils, absolutes, resinoids, resins, concretes etc., but also synthetic materials such as hydrocarbons, alcohols, aldehydes, ketones, ethers, acids, esters, acetals, ketals, nitriles, etc., including saturated and unsaturated compounds, aliphatic, carbocyclic and heterocyclic compounds. Such perfume components are

mentioned, for example, in S. Arctander, Perfume and Flavor Chemicals (Montclair, N.J., 1969), in S. Arctander, Perfume and Flavor Materials of Natural Origin (Elizabeth, N.J., 1960) and in "Flavor and Fragrance Materials-1991", Allured Publishing Co. Wheaton, Ill. USA.

Examples of perfume components are: geraniol, geranyl acetate, linalol, linalyl acetate, tetrahydrolinalol, citronellol, citronellyl acetate, dihydromyrcenol, dihydromyrcenyl acetate, tetrahydromyrcenol, terpineol, terpinyl acetate, nopol, nopyl acetate, 2-phenylethanol, 2-phenylethyl acetate, benzyl alcohol, benzyl acetate, benzyl salicylate, styrallyl acetate, benzyl benzoate, amyl salicylate, dimethylbenzyl carbinol, trichloromethylphenylcarbinyl acetate, p-tert-butyl cyclohexyl acetate, isononyl acetate, vetiveryl acetate, vetiverol, α-hexylcinnamaldehyde, 2-methyl-3-(p-tert-butylphenyl)propanal, 2-methyl-3-(p-isopropylphenyl) propanal, 3-(p-tert-butyl-phenyl)-propanal, tricyclodecenyl acetate, tricyclodecenyl propionate, 4-(4-hydroxy-4-methylpentyl)-3-cyclohexenecarbaldehyde, 4-(4-methyl-3-pentenyl)-3-cyclohexenecarbaldehyde, 4-acetoxy-3-pentyltetrahydropyran, 3-carboxymethyl-2-pentylcyclopentane, 2-n-heptylcyclopentanone, 3-methyl-2-pentyl-2-cyclopentenone, n-decanal, n-dodecanal, 9-decenol-1, phenoxyethyl isobutyrate, phenylacetaldehyde dimethylacetal, phenylacetaldehyde diethylacetal, geranyl nitrile, citronellyl nitrile, cedryl acetate, 3-isocamphylcyclohexanol, cedryl methyl ether, isolongifolanone, aubepine nitrile, aubepine, heliotropin, coumarin, eugenol, vanillin, diphenyl oxidet hydroxycitronellal, ionones, methylionones, isomethylionones, irones, cis-3-hexenol and esters thereof, indan musks tetralin musks isochroman musks macrocyclic ketones, macrolactone musks ethylene brassylate, aromatic nitromusks.

Suitable solvents and diluents for perfumes as mentioned above are, for example, diethyl phthalate, triethyl citrate, ethanol, isopropanol, dipropylene glycol, etc.

The perfume-on-carrier systems (hereinafter for brevity referred to as "POC systems") according to the invention comprise a perfume as defined above and an inert particulate carrier. Inertness of the carrier is tested with a test perfume as described below in the "Perfume Stability Test". For the purposes of this invention a carrier is considered inert if each component in the test perfume 1 of Table 1 below suffers no more than 5% deterioration under the test conditions.

Also the POC systems should easily release a substantial proportion of its perfume content when immersed in a laundry wash liquor. This ability requires the carrier to be sufficiently hydrophobic and the suitability of a carrier can be determined with the "Perfume Release Test" described below. For the purposes of this invention a carrier is considered to pass the test if under the test conditions it is found to release at least 60% of the test perfume 2 of Table 2 below.

The POC systems comprise preferably at least 25% w/w of perfume, more preferably at least 40% and most preferably at least 70% w/w. Yet the POC system should remain free-flowing.

Conveniently the inert carrier particles have a mean diameter in the range 5–50 microns, preferably in the range 10–50 microns, more preferably in the range 15–50 microns, most preferably in the range 20–50 microns. Also, they should preferably have a surface area in the range 100–450 m^2/g, more preferably in the range 100–350m^2/g, most preferably in the range 100–300 m^2/g. Preferably an inorganic carrier is used, such as a silica, zeolite or clay. Typically the carrier particles are of amorphous silica.

In addition to the POC system the concentrates according to the invention may comprise up to 20% w/w of the total perfume quantity, and preferably no more than 10%, in microencapsulated form (e.g. as spray dried starch encapsulates).

The POC systems are conveniently prepared by mixing the perfume with the carrier particles. The mixing of the perfume and the carrier may be carried out in a variety of ways known to the practitioner in the art, for example by spraying the perfume onto the particles contained in a rotary drum or positioned on a conveyor belt. A non-limiting example of a suitable procedure involves the use of a Tatham-Forberg mixer in a low speed, short duration operation. The equipment comprises a twin-drum assembly with a rotary twin-shaft arrangement. Attached to each shaft is a set of carefully profiled paddles positioned at specific angles. The powder charge is fluidised by the paddle rotation and the perfume oil is sprayed on and mixing is continued until perfume take up is complete. The perfumed particle mass is then dropped through the bottom of the mixer into a suitable container.

Absorption or adsorption of perfumes into or onto inert carrier particles will not necessarily protect the perfumes against attack by bleaching compounds. Thus, for use in concentrate formulations containing bleaching agents (which are particularly hostile to perfume components) the perfume is preferably one which is resistant to such attack and retains high performance even when stored in the presence of such hostile ingredients. Suitable non-limiting examples are disclosed in EP 299561 and U.S. Pat. No. 4663068.

The POC systems of the present invention may be incorporated into concentrates using standard powder handling equipment, known in the art, e.g. by utilisation of weigh-belts.

CONCENTRATE FORMULATION EXAMPLES

High non-ionic surfactant content concentrates within the scope of application defined herein are detailed in the references cited above, and two examples are shown below in Tables I and II.

TABLE I

CONCENTRATE "A"	% w/w
Zeolite 4A	33.2
Sodium carbonate	11.2
Dobanol 23-3	12.1
Dobanol 23-6.5	10.2
Sodium LAS	6.2
Sodium sulphate	3.8
Polyacrylate (mw 3000–4000)	3.5
Sodium silicate	1.9
Sodium soap	2.6
POC system particles	2.0
Enzymes	1.3
Anti-redeposition agent	0.9
Antifoam	0.4
Sodium EDTA	0.4
Water, dye, minor components	10.3
	100

TABLE II

CONCENTRATE "B"	% w/w
Zeolite 4A	29.8
Sodium perborate*	15.6
Sodium carbonate	9.7

TABLE II-continued

CONCENTRATE "B"	% w/w
TAED granules**	8.2
Dobanol 23-3	8.7
Dobanol 23-6.5	7.3
Sodium LAS	5.1
Polyacrylate (mw 3000–4000)	3.1
Sodium sulphate	1.6
POC system particles	1.2
Sodium soap	1.5
Sodium silicate	1.5
Enzymes	0.8
Anti-redeposition agent	0.4
Sodium EDTA	0.3
Antifoam	0.3
Water, dye, minor components	4.9
	100

*monohydrate
**tetraacetalylhylene diamine sodium salt

Perfume Stability Test

A particulate sample (2 g) was prepared by incorporating 20% of the perfume test mixture 1 detailed in Table 1 into an inorganic carrier under evaluation. The sample was stored in glass vials (10 ml) for one week at 37° C. prior to analysis. The vials were tightly sealed and free movement of air throughout the test system was minimised by packing the free headspace volume with cotton wool.

Analysis was carried out by extraction of the sample using ethyl acetate, followed by determination of the residual mass of the indicator materials using standard gas chromatographic techniques.

Suitable carriers according to this test are those for which each of the indicator materials suffer no more than 5% degradation (i.e. each component in the system displays at least 95% stability). Such carriers are defined as passing the Perfume Stability Test for the purposes of the present application.

TABLE 1

Perfume Test Mixture 1

Material	w/w %
Benzyl acetate	25
Limonene	25
Tetrahydrolinalol	25
Inert internal standard*	25

*e.g. Tetradecane

Perfume Loading

Equal amounts of carrier material were placed in a series of glass vials and various aliquots of a mixture of perfume components were added (Perfume Test Mixture 2 as shown in Table 2). Each carrier/perfume system was agitated with a spatula, sealed in the vial and stored at ambient temperature for 24 hours prior to inspection. "Perfume Loading" was taken as the loading at which the perfume-on-carrier mixture ceased to flow easily as judged by eye.

TABLE 2

Perfume Test Mixture 2

Material	w/w %
Benzyl acetate	20
Hexylcinnamic aldehyde	20
PTBCHA	20
Tetrahydrolinalol	20
Tonalid	20

KEY: "Tonalid" is a trademark for 1,1,2,4,4,7-hexamethyl-6-acetyl-1,2,3,4-tetrahydronaphthalene
"PTBCHA" is 4-tert.butylcyclohexyl acetate

Perfume Release Test

The POC system under evaluation (containing at least 20% by weight of Perfume Test Mixture 2, Table 2) were equilibrated for 3 days and then added at ambient temperature to an aqueous model wash liquor at a level yielding a theoretical maximum perfume concentration of 40 ppm. The wash liquor (10 g) comprised ca. 0.05% of a mixture of a non-ionic surfactant (Dobanol 25™, a mixture of ethoxylated fatty alcohols with an average of 7 ethoxyl groups) and an anionic surfactant (sodium lauryl sulphate) in a weight ratio of 2.3 to 1.

The mixture was shaken for 10 seconds and immediately placed in a centrifuge and spun for 5 minutes at 2000 RPM.

The bulk of the supernatant liquid was withdrawn (ca. 95% by weight of the aqueous phase, measured accurately) and filtered prior to extraction with ethyl acetate (10 g, containing hexadecane as an internal standard). The ethyl acetate was obtained by centrifugation and was analysed by gas chromatography. The amount of perfume released into the aqueous phase was then calculated.

A perfume release efficiency of 60% or over is taken as indicative of bulk release of the perfume under actual wash conditions where temperatures of 35° C. to 65° C. or higher are common, and longer wash times are likely (e.g. 15–30 minutes, or longer in a soak regime). Carrier materials achieving this are considered to pass the Perfume Release Test.

Carrier material test examples

A range of commercial inorganic particles was subjected to the above tests. The results of these tests together with other relevant data are shown in Tables 3 and 4. It can be seen that the two precipitated silicas meet the criteria specified above, Neosyl GP™ and HP 39™.

TABLE 3

Carrier	Surface Area m^2/g	Mean Particle Size micron	Perfume Loading % w/w	Stability BA	Stability Lim	Stability THL
Gasil 200*	750	4.6	20	95	90	100
Neosyl GP*	200	18	65	95	95	95
Aerosil 200**	200	0.01	80	100	90	100
Zeolite 4A*	—	—	30	85	85	100
Gasil EBN*	320	9	55	100	85	100

TABLE 3-continued

Carrier	Surface Area m²/g	Mean Particle Size micron	Perfume Loading % w/w	Stability BA	Lim	THL
HP39*	300	11	65	95	95	95
Aerosil 300**	300	0.01	75	95	90	100
Neosyl LD*	>400	12	25	90	95	100

Key: BA-Benzyl acetate Lim-Limonene THL-tetrahydrolinalol (i.e. the perfume components of Perfume Test Mixture 1)

TABLE 4

Perfume Release

Carrier	% Perfume released
Aerosil 200**	52
Aerosil R812**	42
Gasil 200**	20
HP39*	70
Neosyl GP*	68

Key: "*" as for Table 4

Application Example 1

POC system particles containing 50% perfume were prepared from Neosyl Gp™ and fragrance "A" as specified in Table 5. These were incorporated into concentrate powder (formulation A, Table 1) at a level of 1.1% to yield a perfume loading of 0.55%. The powder was stored in domestic laminated cardboard containers at 37° C./70% RH (relative humidity) for 4 weeks and examined. No adverse effect was noted on powder flow properties.

Application Example 2

POC system particles containing 50% perfume were prepared from Neosyl GP™ and fragrance "A" as specified in Table 5. These were incorporated into a concentrate powder (formulation B, Table 1) at a level of 1.1% to yield a perfume loading of 0.55%. The powder was stored in domestic laminated carboard containers at 37° C./70% RH (relative humidity) for 4 weeks and examined. No adverse effect was noted on powder flow properties.

Application Example 3

POC system particles containing 55% perfume were prepared were prepared from Neosyl GP™ and fragrance "B" as specified in Table 6. These were incorporated into a concentrate powder (formulation B, Table 1) at a level of 1.45% to yield a perfume loading of 0.8%. The powder was stored in domestic laminated cardboard containers at 37° C./70% RH for 4 weeks and examined. No adverse effects was noted on powder flow properties.

Application Example 4

POC system particles containing 55% perfume were prepared from HP 39™ and fragrance "B" as specified in Table 6. These were incorporated into a concentrate powder (formulation B, Table 1) at a level of 1.4% to yield a perfume loading of 0.8%. The powder was stored in domestic laminated carboard containers at 37° C./70% RH for 4 weeks and examined. No adverse effect was noted on powder flow properties.

Application Example 5

A starch encapsulate was prepared containing 40% perfume oil. The starch was a modified waxy maize starch (Capsul™, National Starch) and the perfume was a freshness accord (Fragrance C, Table 7) of perfume materials susceptible to attack by bleach components in laundry powder. The encapsulate was prepared by normal spray-drying procedures, well known to those experience in the art. An emulsion of 40% starch in water was prepared and sufficient perfume added to constitute 40% of the total of starch and perfume. This emulsion was then spray-dried using a conventional spray-dryer (Niro Mobile Minor) with rotary atomizer, inlet temperature 240° C., outlet temperature 100° C.

POC system particles containing 50% perfume were prepared from Neosyl GP™ and fragrance "B" (see Table 6). These were mixed into a concentrate powder (formulation B, Table 1) at a level of 1.1% to yield a perfume loading of 0.55%. To this product was then mixed in 0.2% of the starch encapsulate detailed above, yielding an overall perfume level of 0.63%. The powder was stored in domestic laminated cardboard containers at 37° C./70% RH for 4 weeks and examined. No adverse effect was noted on powder flow properties.

TABLE 5

Formulation of fragrance "A"

Material	Quantity %
Anther (Q)	1.0
Coumarin	2.0
Gyrane (Q)	0.5
Hexyl cinnamic aldehyde	18.0
Jasmacyclene (Q)	5.0
Jasmopyrane Forte (Q)	4.0
Lilial (G)	10.0
Lixetone (Q)	8.0
Methyl ionone alpha iso (Q)	5.0
4-tert.butylcyclohexyl acetate (Q)	5.0
2-Phenylethyl alcohol	15.0
Pivacyclene (Q)	0.5
Tetrahydrolinalol	6.0
Traseolide (Q)	20.0

Q: trade mark material available from Quest
G: trade mark material available from Givaudan

TABLE 6

Formulation of fragrance "B"

Material	Quantity %
Dimethylbenzyl carbinyl acetate	15.0
4-tert.butylcyclohexyl acetate (Q)	17.5
Tetrahydrolinalol	15.0
Anther (Q)	3.5
Traseolide (Q)	5.0
Decanol	5.0
Phenylpropanol	6.0
Herboxane	10.0
Diphenylmethane	5.0
Cedramber (IFF)	4.0
Hexadecanolide	5.0
2-Phenylethyl alcohol	6.0
Dihydroterpineol	3.0

Q: trade mark material available from Quest
IFF: trade mark material available from IFF

TABLE 7

Formulation of fragrance "C"

Material	Quantity %
Adoxal (G)	1.0
Aldehyde C9 50% in DEP	7.0
Aldehyde C10 50% in DEP	16.0
Aldehyde C11 (undecylenic) 50% in DEP	30.0
Aldehyde C12 50% in DEP	10.0
Methyl nonyl acetaldehyde 50% in DEP	36.0

G: trade mark material supplied by Givaudan
DEP: diethylphthalate (diluent)

We claim:

1. A perfumed, free flowing concentrated laundry detergent power comprising:

at least 15% w/w of detergent surfactants,

less than 10% w/w of fillers,

having a ratio of total solid constituents to total liquid constituents of less than 30:1 and containing at least 0.1% w/w of perfume, wherein the perfume is added as a perfume-on-carrier system consisting of amorphous silica having a mean diameter in the range of 20–50 microns which further have a surface area in the range of 100–450 m^2/gram which carry at least 25% w/w of perfume, which release at least 60% w/w of the perfume in a Perfume Release Test and which are inert as defined by a Perfume Stability Test.

2. A detergent powder according to claim 1, having a bulk density of at least 600 g/l.

3. A detergent powder according to claim 1 or 2 wherein the amount of detergent surfactant is at least 20% w/w and the amount of filler is less than 6% w/w.

4. A detergent powder according to claim 1, wherein at least 35% w/w of the surfactants are liquids.

5. A detergent powder according to claim 4 comprising nonionic and anionic detergent surfactants wherein the ratio of nonionic detergent surfactants to anionic detergent surfactants is at least 3:2 and at least 60% w/w of the nonionic detergent surfactants is liquid.

6. A detergent powder according to claim 4 wherein all detergent surfactants are nonionic of which at least 35% are liquid.

7. A detergent powder according to claim 1 wherein the carrier particles carry at least 40% w/w of perfume.

8. A detergent powder according to claim 7, wherein the carrier particles have a surface area in the range 100–350 m^2/gram.

9. A perfume-on carrier system consisting of amorphous having a mean diameter in the range of 20–50 microns which further have a surface area in the range of 100–450 m^2/gram and which carry at least 25% w/w of perfume, which release at least 60% w/w of the perfume in a Perfume Release Test and which are inert as defined by a Perfume Stability Test.

10. A perfume-on-carrier system according to claim 1, wherein the carrier particles have a surface area in the range 100–350 m^2/gram.

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Bogotá, D.C.,

Abogado
JIMENA ESCOBAR URIBE

ASUNTO	Radicación	05091249
	Trámite	011
	Evento	378
	Actuación	519
	Oficio	862
	Folios	20

Patente de Invención	[x]	Patente de Modelo de Utilidad			[ ]
Vía Nacional	[ ]				
Vía PCT (fase nacional)	[x]	Cap. I	[x]	Cap. II	[ ]

No. Sol. Internacional: PCT/CH2004/000103
Fecha de Presentación Internacional: 2004-02-26

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 le comunica:

1. Documentos estudiados

Capitulo descriptivo: Folios 5 a 14 tal y como fueron presentados.
Capitulo reivindicatorio: Folios 15 a 16 tal y como fueron presentados.
Prioridad: Folio 1 GB0306152.0 19 de marzo de 2003.
Respuesta del solicitante: Folios 80 a 85.
Nuevo capitulo reivindicatorio: Folios 87 a 88.

2. Objeto de la invención

Título de la solicitud: Suministro de Fragancia. Folio 1.

Objeto de la invención: El objeto de la presente solicitud de patente de invención se refiere a un sustrato tal como una tela, que puede ser proporcionado con una fragancia durante el proceso de lavado ó enjuague por la adición al agua de lavado, de una composición que proporciona fragancia sólida de flujo libre que comprende una fragancia depositada en un portador partículado junto con una sal soluble en agua.

El problema técnico planteado en la presente solicitud indica que es deseable suministrar fragancias por medio de su incorporación en productos de lavado. Sin embargo estos contienen tensoactivos que forman micelas y que atrapan las fragancias debido a su hidrofobicidad. Aunque se pueden utilizar fragancias de menor hidrofobicidad, esto restringe el uso de muchas de las fragancias existentes. La solicitud plantea un método de preparación de una composición

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que proporciona fragancia sólida de flujo libre, que consiste de la adición de una fragancia a un material portador particulado en presencia de una sal soluble en agua.

El objeto de la solicitud definido anteriormente se clasificó en la IPC C11D 3/50.

3. Determinación del Estado de la Técnica

Considerando que la fecha de la prioridad de la solicitud internacional PCT es 2003-03-19, se realizó la búsqueda en el estado de la técnica y se encontraron los documentos relacionados a continuación, que pueden afectar la patentabilidad del objeto de la presente solicitud:

Nº	Documento	Título	Fecha de publicación
D5	EP 1061124	Method for preparing fragrance products	2000-12-20
D6	US 5041421	Fragrant material	1991-08-20
D7	US 5840668	Perfumed laundry detergent powders	1998-11-24

4. Análisis de patentabilidad (artículo 14 D486)

4.1. Nivel inventivo *(artículo18 D486)*

La solicitud de patente en estudio presenta *un método* para la preparación de una composición que proporciona fragancia sólida de flujo libre, caracterizada porque comprende *la adición* de una fragancia en la forma de un líquido ó una solución de solvente orgánico a un material portador particulado en presencia de una sal soluble en agua de un metal alcalino ó alcalinotérreo, que comprende la mezcla uniforme de ingredientes secos (portador particulado, sal, cualquier otro componente) seguido por la adición de la fragancia líquida y agitando hasta que se logra un polvo seco de flujo libre, el portador particulado es sílice porosa fina, que contiene opcionalmente hasta 50% en peso de otros materiales particulados absorbentes, donde la cantidad de sal soluble en agua presente en la composición está entre 60% y 85% en peso y la cantidad de portador poroso se encuentra entre 5% y 20% en peso de la composición y la relación de sal soluble en agua a fragancia es desde 20:1 hasta 1,5:1.

Asimismo *una composición* que proporciona fragancia sólida de flujo libre, *caracterizada porque consiste* esencialmente de un *portador particulado en el cual es depositado una fragancia y una sal soluble en agua de un metal alcalino ó alcalinotérreo*, la composición comprende entre 60% y 85% en peso de sal soluble en agua y entre 5% y 20% en peso de un portador particulado, el portador particulado es sílice porosa fina que opcionalmente contiene hasta 50% en peso de otros materiales particulados absorbentes, y la relación de sal soluble en agua a fragancia es desde 20:1 hasta 1,5:1.

El documento D5 divulga sustratos que tienen fragancia prolongada en el tiempo. Específicamente divulga (1) un proceso para la preparación de una matriz de fragancia, donde las fragancias son absorbidas (se encuentran protegidas) en un absorbente sólido y opcionalmente un fijador y (2) la combinación de la matriz de la fragancia con un acondicionador de sustrato y un sustrato.

La matriz de fragancia es un polvo fluido. La proporción de fragancias y fijador de absorbentes sólidos se controlan cuidadosamente para mantener la consistencia de un polvo fluido. Ahora bien los absorbentes sólidos que pueden ser utilizados son, por ejemplo, arcillas, sílices, Zeolitas, sales metálicas, etc.

Por lo tanto D5 ya había divulgado la combinación entre una fragancia y un portador particulado absorbente tipo sílice para obtener un polvo fluido. Lo cual resulta ser equivalente al método y a la composición, planteados en esta solicitud. D5 no indica específicamente la adición de una sal soluble en agua. Sin embargo divulga que el absorbente puede ser una sal de un metal ente otras opciones.

En contraste el documento D6 divulga un material fragante el cual comprende una pluralidad de pellets de sal comprimidos con un aceite aromático disperso dentro de los pellets. Adicionalmente se puede combinar un material higroscópico que puede ser dióxido de silicio, comercialmente conocido como aerosil 200 (columna 3 línea 21). Asimismo el documento D7 divulga la combinación de fragancias con sales solubles en agua y materia absorbentes tales como sílices (columna 5 líneas 57 a 67).

En consecuencia un técnico medio en vista del estado de la técnica en su totalidad es decir, combinando las enseñanzas de los documentos D5 y D6 ó D5 y D7, podría plantear un método y una composición como las acá presentadas, combinando una fragancia con un absorbente tipo sílice para obtener un polvo fluido, que va a ser impregnando en una tela ó sustrato, tal como lo platea D5 y además combinar una sal soluble en agua, como lo divulga cualquiera de los documento D6 ó D7.

Es importante indicar que el uso de sales solubles en agua, es una práctica ampliamente utilizada en la encapsulación y absorción de fragancias. Pero no solo eso cualquiera de los documentos D6 ó D7 ya había divulgado la combinación de un portador poroso tipo sílice, una fragancia y una sal soluble en agua.

De modo tal que el método y la composición de esta solicitud no representan de ningún modo un salto cualitativo en la técnica, no provocan un efecto técnico inesperado ó sorprendente, son evidentes para un técnico medio en la materia y carecen de nivel inventivo.

5. Respuesta a los argumentos del solicitante

Se tuvieron en cuenta los argumentos del solicitante

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6. Conclusión

Los requisitos de patentabilidad de la presente solicitud se encuentran afectados así:

N°	Documento N°	Reivindicaciones afectadas	Requisito que afecta
D5	EP 1061124	1-6	Nivel Inventivo
D6	US 5041421	1-6	Nivel Inventivo
D7	US 5840668	1-6	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 días contados a partir 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.
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 N° 10 de 2001.

ALIX CARMENZA CÉSPEDES DE VERGEL
Directora de Nuevas Creaciones (C)
El anterior requerimiento fue notificado por fijación en lista, de fecha

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CONCEPTO TÉCNICO No. 327	**EXAMINADOR TÉCNICO** Código No.202085