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Señores

Superintendencia de Industria y Comercio
Bogotá D.C.

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
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Eve: 1 REGDEPOSITO
Act: 29 CTOINFORMACION Folios: 1

Cordial saludo,

Yo Vanessa Rosa del Sol Constaín Ordóñez con c.c. 32.141.314. de Medellín en calidad de inventora y solicitante, actualizo mis datos personales para recepción de correspondencia e información referente a la **solicitud de patente de invención No. 03 204.104 con fecha 29 de octubre de 2003.**

Favor enviar la correspondencia e información a la dirección:
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Agradezco su atención,


Vanessa Rosa del Sol Constaín Ordóñez
c.c. 32.141.314 de Medellín

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DIVISION DE NUEVAS CREACIONES

RADICACIÓN EXPEDIENTE No. 03- 204104

EL EXTRACTO DE LA PRESENTE SOLICITUD DE PATENTE DE INVENCION ,
FUE PUBLICADO EN LA GACETA DE PROPIEDAD INDUSTRIAL No 541, DE
FECHA 30 DE JUNIO DE 2004, EL TERMINO HABIL SEÑALADO POR LA LEY
EXPIRA EL 27 DE SEPTIEMBRE DE 2004.

NOTA: Dentro del plazo de los seis (6) meses siguientes a partir de la fecha de publicación en la gaceta de Propiedad Industrial, deberá solicitar y cancelar el examen de patentabilidad so pena de que la solicitud caiga en abandono, de acuerdo con lo establecido en el Artículo 44 de la Decisión 486/2000.

SE PRESENTARON OPOSICIONES POR PARTE DE TERCEROS.

SI _____ **NO** ☒ _____

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

2020

Bogotá, D.C.

Señora
Vanessa Rosa del Sol Constain Ordoñez
Solicitante

Oficio No. 7146

REFERENCIA: Radicación No. 03-204104

Trámite 002

Evento 001

Actuación 430

Folios 011

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.

- Primer capítulo descriptivo: folios 4 al 5
- Segundo capítulo descriptivo: folios 11 al 16
- Capítulo reivindicatorio: folio 17

La solicitud no reivindica prioridad.

2. OBJETO DE LA INVENCION.

Título: "Láminas delgadas de jabón"

La invención se refiere a :

- Producto denominado Láminas delgadas de jabón caracterizado porque se compone por un polímero natural soluble en agua y sales de ácidos grasos que le dan a la lámina además de los usos propios de limpieza, cierta flexibilidad y poder de humectación, siendo totalmente soluble en agua sin dejar residuos. Las láminas delgadas de jabón pueden contener otros aditivos que son comúnmente usados en los jabones. Los aditivos incluyen entre otros: aromas, agentes colorantes, humectantes, antioxidantes.

Clasificación Internacional de Patentes: C11D 13/00

3. CLARIDAD DE LA SOLICITUD.

De acuerdo a la Decisión 486 de la Comisión de la Comunidad Andina en su Artículo 30.- *"Las reivindicaciones definirán la materia que se desea proteger mediante la patente. Deben ser claras y concisas y estar enteramente sustentadas por la descripción"*; se hacen las siguientes observaciones al capítulo reivindicatorio:

En la reivindicación 1 se está presentando un producto que consiste en un jabón en forma de láminas; en términos de patentes todos los productos se deben definir o caracterizar por los componentes que lo constituyen así como por las proporciones en las cuales se encuentran tales componentes. En la reivindicación presentada se puede establecer que está compuesto por un polímero natural, sales de ácidos grasos y el producto puede contar aditivos tales como: aromas, agentes colorantes, humectantes y antioxidantes. Por lo tanto para lograr una definición adecuada del producto se debería incluir en el capítulo reivindicatorio el nombre o los nombres de los compuestos que constituyen el producto, de esta manera se debe indicar el nombre del polímero, la sal o sales de ácidos grasos, los aromas, colorantes, humectantes y antioxidantes. Se debe precisar cada uno de los compuestos que se presentan como constitutivos de la invención; esto con el fin de proporcionar claridad y concisión a la solicitud ya que al presentar el producto de esta manera se está delimitando la materia que se desea proteger. Otro aspecto, que se debe tener en cuenta son las proporciones en las que se encuentra cada uno de los compuestos del producto.

De otra parte, el preámbulo de la reivindicación presentada no se encuentra describiendo el producto de una forma clara ya que se hace mención a: "El producto denominado Láminas delgadas de Jabón"; así como se encuentra presentado el producto no presenta las características técnicas que se podrían encontrar en el estado de la técnica sino que se limita a presentar la denominación del producto.

Se le recuerda al solicitante que una manera adecuada de presentar el capítulo reivindicatorio es por medio de reivindicaciones independientes las cuales contengan las suficientes características técnicas que describan completamente la invención (teniendo en cuenta que deben mantener el preámbulo y la parte característica). Luego se plantean reivindicaciones dependientes en las cuales se presentan características técnicas particulares de la invención con el fin de proporcionarle concisión a la solicitud. También se le recuerda al solicitante que al realizar los cambios al capítulo reivindicatorio, no es posible adicionar materia diferente a la que se encuentra en la presente solicitud.

El título presentado en la solicitud es muy general ya que no indica ninguna particularidad de la invención, por lo tanto al hacer los cambios en el capítulo reivindicatorio es necesario ajustar el título de la solicitud.

Por lo anteriormente expuesto la solicitud no cumple con lo establecido en el art. 30 de la Decisión, se deben realizar los ajustes correspondientes en dicha solicitud.

4. DETERMINACIÓN DEL ESTADO DE LA TÉCNICA

Tomando como base lo anterior y la definición dada en el art. 16.- "Una invención se considerará nueva cuando no está comprendida en el estado de la técnica.

El estado de la técnica comprenderá todo lo que haya sido accesible al público por una descripción escrita u oral, utilización, comercialización o cualquier otro medio antes de la fecha de presentación de la solicitud de patente o, en su caso, de la prioridad reconocida.

Sólo para el efecto de la determinación de la novedad, también se considerará dentro del estado de la técnica, el contenido de una solicitud de patente en trámite ante la oficina nacional competente, cuya fecha de presentación o de prioridad fuese anterior a la fecha de presentación o de prioridad de la solicitud de patente que se estuviese examinando, siempre que dicho contenido esté incluido en la solicitud de fecha anterior cuando ella se publique o hubiese transcurrido el plazo previsto en el artículo 40." , *no obstante la falta de claridad en la solicitud se realizó una búsqueda parcial de anterioridades en los archivos con que cuenta esta Oficina y se encontraron los siguientes documentos relacionados con la materia de la presente solicitud:*

N°	DOCUMENTO	TITULO	FECHA DE PUBLICACION
D1	US 5,062,986	Film-Shape Soap	1991-11-05
D2	US 5,990,047	Toilet bar compositions containing glycolipid surfactants and a process for manufacturing such surfactants	1996-03-26

Los documentos mencionados se anexan al expediente

Una vez se realicen los ajustes del caso, se completará la búsqueda de anterioridades.

7. EVALUACIÓN DE LOS REQUISITOS DE PATENTABILIDAD

El artículo 14 de la Decisión 486 de la Comisión de la Comunidad Andina establece que: "Los Países Miembros otorgarán patentes para las invenciones, sean de producto o de procedimiento, en todos los campos de la tecnología, siempre que sean nuevas, tengan nivel inventivo y sean susceptibles de aplicación industrial."

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

7.1 Evaluación del NIVEL INVENTIVO

De acuerdo al **art. 18.-** *"Se considerará que una invención tiene nivel inventivo, si para una persona del oficio normalmente versada en la materia técnica correspondiente, esa invención no hubiese resultado obvia ni se hubiese derivado de manera evidente del estado de la técnica."*, se conceptúa:

La anterioridad D1 esta presentando un producto que consiste de jabón en forma de láminas delgadas o películas; los componentes de la invención son: un polímero soluble en agua y como un segundo componente jabón. El polímero soluble en agua es poliéster o polieter poliol teniendo un peso molecular promedio mayor de 1000 y un ácido policarboxílico, tiene un peso molecular promedio no menor de 20000. El polímero soluble en agua de esta invención es un poliéster de un polieter poliol y ácido carboxílico. El polímero se obtiene a partir de la mezcla de 10 partes de dietilenglicol y 2.2 partes de dimetil tereftalato y posterior calentamiento a alrededor de 180 a 190 C por una hora. 20 g del polímero obtenido y jabón que contiene sales de sodio de ácidos grasos C18; son amasados a 65 C. La mezcla obtenida fue laminada a 40 C con rodillos para obtener películas de jabón de 50 um.

La anterioridad D2 presenta una composición de jabón en barra que comprende surfactantes glicolípidos y un proceso nuevo para preparar tales surfactantes. Los surfactantes de la invención comprenden uronatos de alquil glicósidos los cuales son de cadena larga; estos compuestos son derivados del ácido D-glucurónico, D-glucurono-6,3- lactona o ácido D- galacturónico. Como una forma preferida de la invención; la composición en barra contiene surfactantes glicolípidos, particularmente sales de sodio de los uronatos descritos anteriormente y los cuales son derivados del ácido D-glucurónico, D-glucurono-6,3- lactona y ácido D-galacturónico. Estos surfactantes glicolípidos aniónicos son considerados como amigables con el medio ambiente por su biodegradabilidad.

Como se puede observar en la anterioridad D1 los productos de jabòn en forma de películas, los cuales son fácilmente portables; ya son conocidos; en este caso particular la composición de tales películas se encuentra constituida por un polímero soluble en agua así como sales de ácidos grasos; componentes que se encuentran en el producto descrito al igual que en el producto presentado en la solicitud.

De otra parte, se encuentran barras de jabòn que contienen compuestos derivados del ácido D-galacturónico, en combinación con sales de ácidos grasos tal como se encuentra presentado por la anterioridad D2 ; por lo tanto es posible establecer que los componentes principales que se encuentran en la solicitud, así como las composición; forman parte de productos que cuentan con el privilegio de patente.

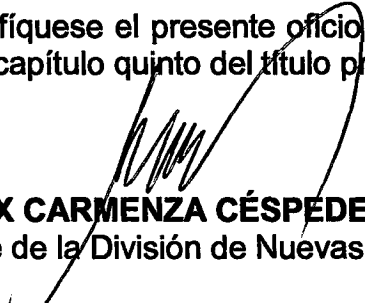
Por lo tanto, la invención presentada es posible derivarla de los productos que se han publicado por medio de los documentos de patente presentados; por lo tanto la solicitud carece de nivel inventivo y no se encuentra cumpliendo con el artículo 18 de la Decisión 486.

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

Nº	DOCUMENTO	REIVINDICACIONES AFECTADAS	REQUISITO QUE AFECTA
D1	US 5,062,986	1	NIVEL INVENTIVO
D2	US 5,501,812	1	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 la notificación de la presente comunicación. **En caso de no dar respuesta al presente requerimiento, o si a pesar de la respuesta subsistieran los impedimentos para la concesión, se denegará la patente.**

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


ALIX CARMENZA CÉSPEDES DE VERGEL
Jefe de la División de Nuevas Creaciones.

El anterior requerimiento fue notificado por fijación en lista, de fecha 14 JUN 2007

CONCEPTO TÉCNICO Nº 0819	EXAMINADOR TÉCNICO CÓDIGO 202064
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United States Patent [19]
Fujita et al.

[11] **Patent Number:** 5,062,986
[45] **Date of Patent:** Nov. 5, 1991

[54] **FILM-SHAPE SOAP.**

[75] **Inventors:** Takeshi Fujita; Chuzo Isoda, both of
Uji; Kazuyo Aketa, Kyoto, all of
Japan

[73] **Assignee:** Dai-Ichi Kogyo Seiyaku Co., Ltd.,
Kyoto, Japan

[21] **Appl. No.:** 505,471

[22] **Filed:** Apr. 6, 1990

[30] **Foreign Application Priority Data**

Apr. 20, 1989 [JP] Japan 1-101163

[51] **Int. Cl.³** C11D 17/06; C11D 7/04

[52] **U.S. Cl.** 252/134; 252/108;
252/174; 252/174.21; 252/174.23; 252/DIG.
16

[58] **Field of Search** 252/134, 174, 174.23,
252/DIG. 16, 108, 174.21

[56] **References Cited**

U.S. PATENT DOCUMENTS

3,293,684 12/1966 Tundermann 252/92

FOREIGN PATENT DOCUMENTS

55-133497 10/1980 Japan 252/174
0165998 12/1980 Japan 252/134
62-43497 2/1987 Japan 252/134

Primary Examiner—Paul Lieberman

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Attorney, Agent, or Firm—Stevens, Davis, Miller &
Mosher

[57]

ABSTRACT

The present invention relates to a film-shape soap comprising (a) a water soluble polymer and (b) a soap; said water soluble polymer is a polyester of polyether polyol having an average molecular weight of more than about 1,000 and a polycarboxylic acid, and has a weight average molecular weight of not less than about 20,000, which has a rapid soluble time in water in spite of a lower equilibrium moisture content, excellent washability, little stimulation to a skin, flexible (not brittle) and stable properties, and is suitable for a portable type toilet soap, disposable soap and the like.

5 Claims, No Drawings

FILM-SHAPE SOAP

BACKGROUND OF THE INVENTION

The present invention relates to a film-shape soap.

It is 60 years since a film-shape soap, so called "paper soap" was known, but the classical paper soap was too brittle to be practically used.

Recently, the demand of a film-shape soap has become increased for portable use, disposable use, unit usage, and so on, especially as the sanitary sense has become more popular. In order to improve the brittleness of the classical paper soap it has been tried the addition of various kinds of a film formable polymer material into the soap. Most of these trials, however, have been failed due to the incompatibility of the soap with a conventional film formable polymer material and the high melting point of the soap itself.

Japanese Patent Application KOKAI No.53-91912 discloses a film-shape detergent using a synthetic detergent which is comparatively easy to prepare a film instead of soap and methyl cellulose as a film forming material. For this film-shape detergent obtained, however, have been pointed out many defects attributed to the synthetic detergent such as the cause of hand chapping, poor solubility in water, hard or stimulus feeling to hand in use and so on. Therefore, it has been desired the development of a film-shape soap using a soap itself which is mild to a skin, and has no stimulation.

SUMMARY OF THE INVENTION

The present invention provides a film-shape soap, which is useful for portable use, disposable use, unit usage and the like. The film-shape soap of the present invention comprising (a) a water soluble polymer and (b) a soap; said water soluble polymer is a polyester of polyether polyol having a weight average molecular weight of more than about 1,000 and a polycarboxylic acid, and has a weight average molecular weight of not less than about 20,000.

The polymer used in the present invention has an excellent compatibility with a soap and can give a thin layer soap when mixed with a soap.

DETAILED DESCRIPTION OF THE INVENTION

The present invention relates to a film-shape soap which comprises (a) a water soluble polymer and (b) a soap; said water soluble polymer is a polyester of polyether polyol having a weight average molecular weight of more than about 1,000 and a polycarboxylic acid, and has a weight average molecular weight of not less than about 20,000.

The water soluble polymer of the present invention is a polyester of a polyether polyol and a polycarboxylic acid.

The polyether polyol used for the production of the polyester is a polyol which can be prepared by the addition of an alkylene oxide to a compound having two active hydrogen atoms in a molecule (referred to as an active hydrogen-containing compound hereinafter). There are exemplified as an alkylene oxide ethylene oxide, propylene oxide, butylene oxide and the like. Mixture of the alkylene oxide may be used. In order to furnish the polyester with a sufficient water solubility it is preferable to use ethylene oxide partially as an alkylene oxide, and preferably more than about 70 percent by weight of the total alkylene oxide. The active hydro-

gen-containing compound may include alkylene glycols, such as ethylene glycol, propylene glycol, butylene glycol, hexylene glycol and the like; aromatic polyols such as hydroquinone and the like; amines such as ethylamine, hexylamine, cyclohexylamine, benzylamine, aniline, imidazolidine, and the like; amides; mercaptans; and so on. In case the active hydrogen-containing compound contains hetero atoms such as amines, amides or mercaptans, the obtained compound is also included in the conception of "a polyether polyol", though containing the hetero atoms in the molecule.

A preferable polyether polyol is a polyether diol, in the aspect of solubility in water and polymeric ability. A monoalcohol cannot extend the polymer chain, and a triol or a polyol having hydroxyl groups more than 3 is tend to be reticulated and to be insoluble in water.

The polyether polyol itself may contain several kinds of oxyalkylene moieties such as an oxyethylene moiety and an oxypropylene moiety, which may be a block copolymer or a random copolymer.

The polyether polyol can be obtained according to a conventional method, for example, reacting a desirable alkylene oxide or a mixture of two or more kinds of alkylene oxide with two active hydrogen-containing compound(s) in the presence of catalyst, for instance, alkaline catalyst such as potassium hydroxide, sodium carbonate or acid catalyst such as boron trifluoride under a suitable pressure such as 0-10 atoms (gauge pressure) at a temperature of, for example, from about 60° to 160° C.

Alkaline catalyst for an addition polymerization of the alkylene oxide is especially preferable, because the alkaline catalyst can be also used as a catalyst for a successive esterification without any elimination process of the catalyst or use of additional catalyst.

The content of the polyoxyethylene moiety in the polyether polyol is preferably more than 50 % by weight, more preferably 70-100 % by weight based on the total weight of the polyether polyol. If the content of the polyoxyethylene moiety is less than 50 %, the water-solubility becomes insufficient.

The polyether polyol should be water soluble, and preferably has a weight average molecular weight of more than about 1,000, more preferably more than about 2,000, most preferably about 3000-50,000 in the aspect of the water solubility and the tear strength.

As aforementioned, the most preferable examples of the polymers for the present invention are polyesters of polyether polyol with polycarboxylic acid. Such polymers can be prepared according to Japanese Patent Application KOKAI No. 56-226018, which is incorporated into the present specification.

Examples of the polycarboxylic acid usable for the preparation of the polyester are aliphatic polycarboxylic acid such as malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, maleic acid, fumaric acid, mesaconic acid, malic acid, tartaric acid, hexane tricarboxylic acid, and the like; aromatic polycarboxylic acid such as Phthalic acid, isophthalic acid, terephthalic acid, trimellitic acid, pyromellitic acid, 1,5-naphthalenedicarboxylic acid, dimmer acid, and the like. The polycarboxylic acid may be a partially or wholly ester, which can be reacted by ester interchanging reaction, or may be an acid anhydride such as maleic anhydride, phthalic anhydride, pyromellitic dianhydride, and the like. Lower alkyl esters of the polycarboxylic acid are also usable, such as

methyl ester, polymethyl ester, mono or polyethyl ester, and the like.

Mixture of the polycarboxylic acid may be used.

Most preferable polycarboxylic acids are phthalic acid, terephthalic acid, isophthalic acid, sebacic acid, pyromellitic acid; lower alkyl esters thereof such as dimethyl esters, diethyl esters; anhydride such as pyromellitic dianhydride and the like.

A preferable weight average molecular weight of the polymer such as polyesters of the polyether polyols and the polycarboxylic acid is more than about 10,000 - 500,000. If the molecular weight is less than 10,000, the water-solubility of an obtained polymer film or sheet would be sufficient, but the tear strength will lower and the equilibrium moisture content will exceed the lower limit required, whereas the molecular weight of more than 500,000 will decrease the water-solubility.

The ratio of the polyether polyol and the polycarboxylic acid, ester or acid anhydride is not restrictive so far as the molecular weight of the polyether polyol is not less than 1,000, and that of the obtained polymer is not less than 10,000 and not more than 500,000 is satisfied.

The esterification of the polyether polyol and the polycarboxylic acid, ester, or acid anhydride can be effected by a conventional manner, for example, by heating under reduced pressure in the presence of catalyst such as p-toluene sulfonic acid, boron trifluoride, potassium hydroxide, sodium hydroxide, magnesium hydroxide, alkali methals and the like.

Preferable temperature is about 80°-250 ° C., and preferable pressure is about 0.001-20 Torr. Reaction can be usually completed within 0.5-10 hours. Of course, the reaction may be carried out as removing generated water or lower alcohol by the ventilation of nitrogen gas. But the esterification aforementioned is only a suitable example, and should not be construed restrictively.

The molecular weight may be controlled by the ratio of the polyether polyol and the polycarboxylic acid, or by controlling the reduced pressure. The latter is more practical and easy. As the reaction pressure is lower, a polymer having a larger molecular weight can be obtained.

Though the aforementioned polyesters are most suitable polymers for providing a film-shape soap due to its compatibility with a soap, another water soluble polymers such as methyl cellulose, hydroxyethyl cellulose, hydroxypropyl methyl cellulose, carboxy methyl cellulose, polyvinyl alcohol, polyvinylpyrrolidone, polyacrylates (e.g. sodium, potassium or ammonium salts) and the like may be mixed therewith. However, as the polymer of the present invention is immiscible with polyvinyl alcohol, and the co-use with cellulose derivatives makes the film highly water-absorbable, the content of another water soluble polymer is preferably less than 10 % by weight of the polymer of the present invention.

A soap which may be used in the present invention is not restricted. Any kinds of fatty acid salts usable for a soap, for instance, a body soap which is directly applied to washing a humane body such as a toilet soap, a bath soap, a medicated soap, a deodorant soap and the like; a laundry soap; a domestic soap such as detergent for a washing machine, for a kitchen, and the like; and a soap for industrial use.

As a fatty acid for the soap may be exemplified a saturated or unsaturated fatty acid, a branched or straight fatty acid or mixture thereof. The fatty acid preferably includes lauric acid, myristic acid, palmitic

acid, stearic acid, arachidic acid, oleic acid, elaidic acid, erucic acid, linoleic acid, linolenic acid, ricinoleic acid, 12-hydroxy stearic acid and the like.

The alkali used for forming the soap includes alkaline metal such as sodium, potassium, ammonium, alkanolamines and the like. Most preferable one is sodium.

The soap may be produced according to a conventional process, for instance, by saponification of an oil or a fat such as tallow oil, palm oil, palm kernel oil, a coconut oil, castor oil, lard, hydrogenated fish oil, hydrogenated tallow, and sometimes, cottonseed oil, soybean oil, peanut oil, olive oil, and the like, most suitable oil is a mixture of tallow and coconut oil; or neutralizing a fatty acid with a suitable alkali.

The weight ratio of the water soluble polymer to the soap may be preferably from about 10/90 to about 80/20, more preferably about 20/80 to about 60/40 in terms of solid.

The content of water in the mixture of the water soluble polymer and the soap may be about 1-about 40 percent by weight, more preferably about 5-about 25 percent by weight based on the total weight of water, water soluble polymer and the soap.

The film-shape soap of the present invention may contain other additives which is usually used in a soap. The additives include a perfume, a deodorant (e.g. phenols, cresols, bisphenols, salicylic acid and the like) a coloring agent, a preservative, a bactericide, a humectant, a plasticizer, an antioxidant, a superfatting agent (an oily material to prevent a skin from excess removal of a fat thereon, e.g. a higher fatty acid, a higher fatty alcohol, lanolin, squalane and the like; or to protect the skin instead of the fat removed therefrom, e.g. a glycerin, glycol, diethylene glycol, lecithin and the like), water softening agents such as chelating agents, lime soap dispersing agents, builders, transparents such as glycerin, rosin soap, sugar, alcohol and the like, redeposition preventing agents such as carboxymethylcellulose, foam stabilizers, defoamers and the like.

The film-shape soap of the present invention may contain other water soluble polymers such as methyl cellulose, hydroxypropyl methyl cellulose, carboxymethylcellulose, hydroxyethyl methyl cellulose, hydroxybutyl methyl cellulose, hydroxy ethyl cellulose, hydroxy propyl cellulose, polyacrylate, pullulan, polyvinyl alcohol, and the like; and additional surfactants other than a soap. In case that the film-shape soap is for a body soap such as a toilet soap, a bath soap, a deodorant soap, and the like, which is most suitable one for the object of the present invention, the content of the additional surfactant prefer as little as possible in most case, but sometimes the addition of a surfactant other than a soap improves the washability, foaming property, soft feeling of a skin, prevention of stimulation and the like. Typical surfactant which may added to the film-shape soap of the present invention is an alkyl ester of α -sulf fatty acid salt, an N-acylglutaminic acid salt, alkyl sulfate, alkylbenzenesulfonate, fatty acid isopropanolamide sulfonate, α -glyceryl monoalkylether- α' -sulfonate, α -acyl- α' -sulfonyldiglyceride, acyl-N-methyltauride, polyoxyethylene alkyl ether, polyoxyethylene alkyl phenol ether, polyoxyethylene alkyl ester, polyoxyethylene alkyl ether sulfate and the like.

The film-shape soap of the present invention may be prepared by kneading a water soluble polymer as aforementioned and soap, and other additives if necessary, and then the mixture is shaped like a film or a sheet (in the present specification both of such a film or a sheet

are referred to as a film altogether). In this process it is preferable to use a small shape soap such as powder, granule, pellet, flake, needle and the like. As a conventional kneader, extruder, roll and the like can be used. The process for kneading may be carried out at a temperature that the water soluble polymer and the soap are softened, for instance, about 30°-200° C., more preferably about 50° C. to 90° C. for about 0.5-1 hour. Of course, the temperature is not restricted to the above, because the melting point of the soap is depended on the kind of salt, the chain length of fatty acid, the content of unsaturated fatty acid, water content and the like. If a soap having a comparatively high moisture content such as 30 percent by weight, the temperature may be lower than 50° C.

As a means for shaping a film or sheet rolling extrusion from a T-die, flow-extending and the like are exemplified. The thickness of the film-shape soap of the present invention can be from 10 μ m to 500 μ m preferably 20 μ m to 100 μ m.

The film-shape soap of the present invention can be controlled in the thickness, softness, water solubility, tear strength, equilibrium moisture content, soluble time and so on by controlling the water content in the film-shape soap, the ratio of the soap and the water soluble polymer, water solubility of the polymer, kneading condition, kinds of the soap and the polymer, and the like.

According to the present invention a flexible film-shape soap has a high tear strength and a small equilibrium moisture content, which can be easily and rapidly dissolved in water. Therefore, the film-shape soap of the present invention may be used as a portable toilet soap, a disposable soap, a soap for unit usage by cutting the film-shape soap to a suitable size or rolling it. The film-shape soap may be used as a rolling film type.

Illustrating the invention are the following examples, which, however, are not be construed as limiting the invention to their details. All parts and percentages in the examples, as well as throughout this specification are by weight unless otherwise specified.

EXAMPLE 1

Polyethylene glycol (an weight average molecular weight of 10,000) 100 parts and dimethyl terephthalate 2.2 parts were mixed, and heated at about 180° to 190° C. for one hour in the presence of catalytic amount of potassium hydroxide as removing produced ethyl alcohol under a reduced pressure of about 0.1 Torr to give a polyester having an average molecular weight of about 130,000 (this compound is referred to as a Polymer A hereinafter).

The Polymer A 20 g and a toilet soap powder which contains sodium salt of C₁₈-fatty acid as a main ingredient (water content: 20 % by weight) 20 g were kneaded at 65° C. for 20 minutes in a twin-roll mill. The mixture obtained was rolled at 40° C. by rolling to give a film-shape soap of 50 μ m.

Hand washing test using the film-shape soap prepared in the above process was made by a panel consisting of ten members. Nine members in the panel evaluated that the film-shape soap was equal to the soap, a row material in the washability, and all members in the panel recognized that no insoluble material remained on the hands, and were impressed with an excellent defoamable feeling, moist and refreshing on the skin.

The film has a flexible and a comparatively high tear strength, and is not brittle. After the film was held at 35° C. and RH 90 % for one month, the appearance, weight, physical properties (tear strength and water solubility) were not changed. This means that the compatibility of the water soluble polymer of the present

invention and the soap is excellent and soap does not bleed out; the polymer is not hydrolyzed with the alkali of the soap; the film-shape soap of the present invention has little absorbability of moisture in atmosphere, but high water solubility when it is immersed into water. Accordingly, the film-shape soap of the present invention is suitable for a portable toilet soap.

EXAMPLE 2

Ethylene oxide 1,900 parts was polymerized onto polypropylene glycol (weight average molecular weight of about 2,000) 100 parts according a usual manner.

The obtained polyether polyol was esterified with dimethyl sebacate 20 parts according to a similar manner as in Example 1 to give a polyester having a weight average molecular weight of 200,000 (referred to as Polymer B hereinafter).

The Polymer B 15 g was mixed with an industrial soap (Genbu Marseilles Sekken ®) (sodium salt of tallow fatty acid as a main ingredient): available from Dai-Ichi Kogyo Seiyaku Co., Ltd.), a needle like material, 25 g was roughly mixed, and then the mixture was charged into a small extruding through a kneading zone kept at 65° C., and passed through a T die to give a film-shape soap of 70 μ m thickness.

The above film-shape soap was evaluated by ten members in a panel according to similar manner as in Example 1. All members answered that the washability and the feeling during and after the use of the film-shape soap were not inferior to the soap itself used as a raw material.

COMPARATIVE EXAMPLE

A soap used in the Example 1 50 g was mixed with an aqueous solution of methyl cellulose (2% solution) 100 g at 30° C., and flowed on a glass plate. The mixture on the glass plate was dried at 100° C. to give a film-shape soap of soap content of about 60 percent by weight and a thickness of about 50 μ m.

This film-shape soap contains bubbles therein due to foams generating at the film production.

The above film soap contains bubbles therein due to foams generating at the film production.

The above film-shape soap was evaluated by ten members in a panel according to similar manner as in Example 1. All members answered that the film was hard, brittle, worse solubility and worse feeling or touch at use and after use.

What is claimed is:

1. A film-shape soap which comprises (a) a water soluble polymer and (b) a soap; said water soluble polymer is a polyester of polyether polyol having an weight average molecular weight of more than about 1,000 and a polycarboxylic acid, and has a weight average molecular weight of not less than about 20,000.

2. A film-shape soap of the claim 1, in which the polymer is a polyester having a polyoxyethylene moiety.

3. A film-shape soap of the claim 1, in which the polymer is an ester of polyalkylene glycol having a weight average molecular weight of about 1000 to 50000 and polycarboxylic acid selected from the group consisting of terephthalic acid, phthalic acid, isophthalic acid, sebacic acid and pyromellitic acid.

4. A film-shape soap of the claim 1, in which the ratio of the water soluble polymer and the soap is from about 20:80 to about 60:40 by weight in terms of solid.

5. A film-shape soap of the claim 1, in which the film has a thickness of from about 10 μ m to 500 μ m.

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US005501812A

United States Patent [19]

Vermeer et al.

[11] **Patent Number:** 5,501,812[45] **Date of Patent:** Mar. 26, 1996

[54] **TOILET BAR COMPOSITIONS CONTAINING GLYCOLIPID SURFACTANTS AND A PROCESS FOR MANUFACTURING SUCH SURFACTANTS**

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[21] **Appl. No.:** 135,237

[22] **Filed:** Oct. 12, 1993

Related U.S. Application Data

[63] **Continuation-in-part of Ser. No. 816,421, Dec. 31, 1991, abandoned.**

[51] **Int. Cl.⁶** C11D 1/18; C11D 1/38; C11D 3/26; C11D 7/32

[52] **U.S. Cl.** 252/174.17; 252/547; 252/DIG. 5; 252/DIG. 6; 252/DIG. 14

[58] **Field of Search** 252/174.17, DIG. 5, 252/DIG. 6, DIG. 14, 547; 536/18.5, 58

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[57]

ABSTRACT

The present invention relates to toilet bar soap compositions comprising 1 to 45% by wt. of specified alkali metal (alkyl glycosid) uronates and to processes for making them.

17 Claims, No Drawings

Exp. 03 - 204 104.

TOILET BAR COMPOSITIONS CONTAINING GLYCOLIPID SURFACTANTS AND A PROCESS FOR MANUFACTURING SUCH SURFACTANTS

This is a continuation-in-part of application Ser. No. 07/816,421, filed on Dec. 31, 1991, now abandoned.

FIELD OF INVENTION

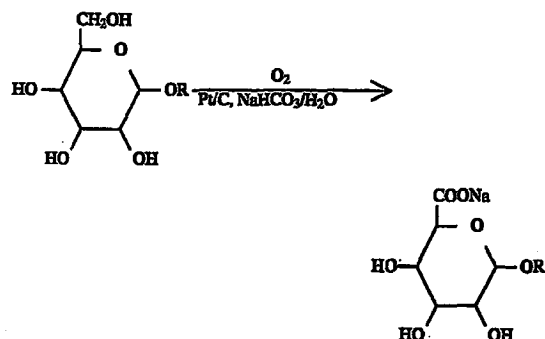
This invention relates to an unexpectedly mild novel toilet bar composition comprising glycolipid surfactants and a new process for preparing such surfactants. The surfactants of the invention are long chained (alkyl glycosid)uronates derived from D-glucuronic acid, D-glucurono-6,3-lactone or D-galacturonic acid.

This invention is also concerned with the preparation of these (alkyl glycosid)uronates having a commercially feasible economical output and with the preparation of these (alkyl glycosid)uronates of high purity and good color. The (alkyl glycosid)uronates are prepared without hydroxyl group protection and without oligomerisation or polymerization.

BACKGROUND OF THE INVENTION

The demand for mild, environmentally friendly surfactants has been steadily rising. In general, most toilet bar compositions contain surfactants based on petro chemicals. Since these materials often have handling, storage and environmental hazards associated with them, it would be most desirable to use surfactants which are instead derived from agriculturally grown materials, such as carbohydrates. These naturally occurring compounds represent a source of renewable raw materials that are readily available, inexpensive, biodegradable, aquatically favorable and optically pure.

(Alkyl glycosid)uronates are known to be prepared by catalytic air oxidation of alkyl glycosides over a platinum or palladium catalyst in alkaline aqueous solution according to the following equation:



U.S. Pat. No. 2,562,200 to Mehlretter and U.S. Pat. No. 2,845,439 to Corn Products Refining Company, both teach the above identified process using short chained sodium(alkyl glycosid)uronates in which R is methyl, ethyl or hydroxyethyl. European Patent No. 0,326,673 and German Patent No. 3,803,465 to Ripke et al., also teach the above-identified process which is directed towards long chained sodium(alkyl glycosid)uronates in which R is greater than 8 carbons.

These known processes suffer from the distinct disadvantage of being performed under dilute reaction conditions (3 to 6% alkyl glycoside in 85 to 97% water) resulting in low product output and therefore rendering the process uneconomical. In fact, higher concentrations of alkyl glycoside (>10%) cause a retardation of reaction velocity, and yields are diminished (K. Heyms and H. Paulsen, Adv. Carbohydr. Chem., [1962], V. 17, p. 176).

In addition to failing to teach the specific process of the invention, none of these references or other prior art of which applicant is aware of teaches or suggests the use of these surfactants in toilet bar compositions.

More specifically, while Ripke 3,803,465 or EP 0,326,672 suggests that the compounds of the invention may be suited for detergent or cleanser applications, however, there is no suggestion to use them in toilet bar compositions. It must be recalled that the surfactants which are anionic would be classified as generally being harsh to the skin. Thus, while one of ordinary skill in the art might contemplate use of these anionics in detergent compositions where there would be no real direct contact with the skin, such a person would be especially disinclined to formulate the anionic surfactant in a toilet bar where the bar is intimately rubbed by the consumer against the skin. It is only after applicants ran zein tests and discovered the surfactant to be relatively mild on skin that it might even dawn to one of ordinary skill in the art to formulate the surfactant in a bar. Further, the data present in the subject specification cannot be used in making such an assessment.

In the parent of the subject application, the Examiner cited a washing cleanser patent (U.S. Pat. No. 5,132,037) in combination with several chemical abstract references relating to uses of uronic acids. On particular, the Examiner noted CA 101:97490Z, relating to the use of uronic acid for smoothing skin and eliminating flaking. The chemical abstract references, however, all relate to uronic acids, not surfactant derivatives. It is anionic surfactants which are known to be harsh and there would have been no motivation, let alone a teaching, to prepare a surfactant from the acid.

Finally, WO 9302092 to Zschimmer & Schwarz teach galacturonic acid derivatives similar to the compounds of the invention. This application was published after the filing date of the priority date to which the subject application is entitled and is not available as prior art.

Accordingly, it is one object of the invention to provide a novel toilet bar composition comprising the surfactants of the invention.

Another object of the invention is to provide such a bar that is mild to the skin and has good lathering and tactile properties.

It is yet another object of the invention to provide a new and improved process for the manufacture of (alkyl glycosid)uronates, preferably alkali metal (alkyl glycosid)uronates such as sodium(alkyl glycosid)uronates.

It is a particular object of the invention to prepare alkali metal (alkyl glycosid)uronates in good yield, high purity and desirable color within a commercially feasible economical output.

These and other objects of the invention will become more apparent from the detailed description and examples that follow.

SUMMARY OF THE INVENTION

One embodiment of the invention relates to a surprising mild novel bar composition containing glycolipid surfac-

tants, particularly long chained sodium (alkyl glycosid)uronates derived from D-glucuronic acid, D-glucurono-6,3-lactone and D-galacturonic acid.

The second embodiment of the invention relates to a new process for preparing such surfactants. The process is an improvement over the art known process for the preparation of sodium(alkyl glycosid)uronates, wherein the improvement comprises reacting a uronic acid directly or nondirectly with a long chained alcohol in presence of an acid catalyst followed by alkaline hydrolysis containing a bleaching agent. This embodiment of the invention is particularly directed to preparing (alkyl glycosid)uronates, preferably alkali metal (alkyl glycosid)uronates, in good yield, high purity and desirable color within a commercially feasible economical output. The (alkyl glycosid)uronates of the invention have surfactant properties equal to, or better than other well known anionic surfactants based on petrochemicals, thereby indicating that they are viable, environmentally sound alternatives to traditional petrochemical surfactants.

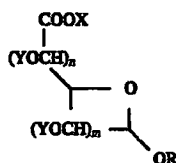
DETAILED DESCRIPTION OF THE INVENTION

The present invention relates to a class of environmentally friendly "green" anionic glycolipid surfactants. In particular, the invention relates to (alkyl glycosid)uronates derived from D-glucuronic acid, D-glucurono-6,3-lactone or D-galacturonic acid.

In one embodiment of the invention, an unexpectedly mild novel toilet bar composition containing sodium(alkyl glycosid)uronate is described.

In a second embodiment of the invention, a new and improved process for the manufacture of (alkyl glycosid)uronates is described.

In general, (alkyl glycosid)uronates are defined as glycosides of uronic acid, uronic acid salts or uronic acid lactones of the following formula:



wherein:

$n=0-2$, preferably $0-1$;

$m=2-4$, preferably $2-3$;

Y is a hydrogen atom, mono-, oligo- or polysaccharide; uronic acid, uronic acid salt, uronic acid lactone or polyuronic acid;

R is a straight or branched chain alkyl or alkenyl group which may contain an aromatic, cycloaliphatic or polyalkoxyalkyl radical comprising 8 or more carbons;

X is a hydrogen atom, alkali metal, alkaline earth metal, ammonium, mono-, di- or trialkanol ammonium group within 2-3 carbon atoms; alkyl-substituted ammonium group with 1-5 carbon atoms; or basic amino acid.

Suitable mono-, oligo- or polysaccharides that may be used to form sodium (alkyl glycosid)uronates of the invention include, but are not limited to glucose, galactose, mannose, gulose, sucrose, lactose, fructose, sorbitol, maltose and starch.

Examples of polyuronic acids, uronic acids or their lactones which may be used to form sodium(alkyl glycosid)uronates of the invention include, but are not limited to

D-glucuronic acid, D-glucurono-6,3-lactone and D-galacturonic acid. Other examples include D-mannuronic acid, L-guluronic acid, L-lyxuronic acid, L-iduronic acid, pectin, algin, alginic acid, oxidized starch, oxidized cellulose and acacia.

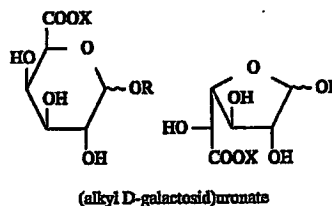
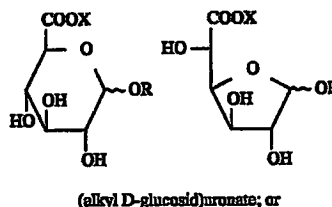
Suitable aliphatic hydrocarbon radicals include saturated and unsaturated radicals including but not limited to methyl, ethyl, amyl, hexyl, heptyl, nonyl, decyl, undecyl, dodecyl, tridecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl and octadecyl; allyl, decenyl, dodecenyl, tetradecenyl, oleyl, linoleyl and linolenyl. The active compounds of the invention may contain straight or branched aliphatic groups. Aromatic radicals are exemplified by benzyl, aniline or substituted benzyl or aniline groups. Suitable mixed aliphatic aromatic groups are exemplified by benzylpropyl, phenylethyl, phenoxyethyl and vinylbenzyl. Cycloaliphatic radicals are exemplified by but not limited to cyclopentyl and cyclohexyl.

Suitable alkali metal, alkaline earth metal, ammonium, alkanol ammonium groups include, but are not limited to sodium, potassium, lithium, methylamine, dimethylamine, trimethylamine, ammonia, methylglucamine, glucamine, monoethanolamine, triethanolamine, 2-amino-2-ethyl-1,3-propanediol and chitosamine.

Suitable basic amino acid groups include alkaline salts of glycine, alanine, valine, leucine, serine, lysine, aspartic acid, methionine and glutamine. The (alkyl glycosid)uronates may also be ethoxylated, propoxylated or mixtures thereof.

The chemical behavior of sodium(alkyl glycosid)uronates prepared by the process of the invention results in the formation of pyranosides, furanosides or preferably both.

In one embodiment of the invention, an alkali metal (alkyl glycosid)uronate is used as a surfactant in a toilet bar composition. Examples of specific (alkyl glycosid)uronates are set forth below:



wherein, in each case, R is a substituted or unsubstituted, saturated or unsaturated alkyl group having 8-24 carbons, preferably 10-16 carbons. R may also be an alkoxyalkyl chain. X may be sodium, potassium, ammonium, alkanolammonium group, or basic amino acid, however sodium is preferred.

In one embodiment of the invention, an unexpectedly mild novel toilet bar composition containing (alkyl glycosid)uronate as the sole surfactant or cosurfactant is described.

Typical toilet bar compositions are those comprising fatty acid soaps used in combination with a detergent and free fatty acids. It should be noted that the composition may comprise no fatty acid soap and may be based on actives