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Señora Jefe de la
DIVISION DE NUEVAS CREACIONES
Superintendencia de Industria y Comercio
E S D

EDIFICIO S
CARRERA 4 No
BOGOTÁ 8 CO

SUPERINDUSTRIA Y COMERCIO
Radicacion : 00064176 00000003
Fecha (AND): 2002-12-20 17:11:58
Tramite : 002 PATENTES D 1 REGISTRO/ 538 PETICION
Dependencia: 2020 DIVISION DE NUEVAS CREACIONES
Folios: 2
(1) 347 3611
) 310 4995

TRÁMITE : 002

EVENTO : 001

ACTUACIÓN : 538

Referencia Expediente No 00-064.176

Asunto Solicitud de patente para ESTRUCTURA
ABSORBENTE EN UN ARTÍCULO ABSORBENTE Y
METODO DE PRODUCCION DE ESTE

Solicitante SCA HYGIENE PRODUCTS AB

Fecha de solicitud 28 de agosto de 2000

Publicada en la Gaceta No 517 del 27 de Junio 2002

1 Yo, Emilio FERRERO, identificado como aparece al pie de mi firma, actuando como apoderado en el asunto de la referencia, atentamente, solicito se efectúe el examen de patentabilidad de la solicitud en referencia según lo establecido en el artículo 44 de la Decisión 486 de la Comisión de la Comunidad Andina Para tal efecto presento junto con este escrito el recibo No 02-80,899 por valor de \$318 000 00, que corresponde a la tasa fijada según lo dispuesto en la resolución No 44691 del 31 de diciembre de 2001

2 Ruego atentamente a la Señora Jefe se sirva ordenar que se anexe este recibo, para que se efectúe dicho examen y se continúe con el trámite de la solicitud

Bogotá, 20 DIC 2002
Señora Jefe,


EMILIO FERRERO
T P A No 31 929

COLO 11256 801 042 8
JBP/JFR/ID-000668/WPC11256



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Industria y Comercio

SUPERINDUSTRIA Y COMERCIO
Radicación : 00064174 00000004 Folios: 2 N C I A
Fecha (AMD): 2002-12-20 17:11:13
Trámite : 002 PATENTES D 1 REGISTRO/ 538 PETICION
Dependencia: 2020 DIVISION DE NUEVAS CREACIONES

NIT : 800176089-2

RECIBO OFICIAL DE CAJA : 02 - 80,899
FECHA : DICIEMBRE 2 DE 2002

***** CONSIGNACION *****

DEPOSITANTE	TIPO PAGO	BANCO	CUENTA	No. PAGO	FECHA PGO	Vr PAGO
CAVELIER ABOGADOS	CONSIGNACION	BANCO POPULAR	050-00110-6	7030558	02/12/2002	28,771,000 00

***** CONCEPTO *****

CANT.	RENTISTICO	CONCEPTO	TOTAL CONCEPTO
1	50005-01-01 SOLICITUDES	112 PTC CAP-1 EXAMEN DE PATENTIBILIDA	318,000 00
		TOTAL :	\$ 318,000.00

SON TRESCIENTOS DIEZ Y OCHO MIL PESOS

RESPONSABLE • 

RECIBO DE CAJA APLICADO AL EXPEDIENTE No. _____

060 11256-001-042-8

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

2020
Bogota, D C , 16 de mayo de 2005

Doctor
EMILIO FERRERO
Apoderado

Oficio No 00-12804

ASUNTO	Radicacion	00-64176
	Tramite	002
	Evento	001
	Actuación	430
	Folios	013

Como resultado del examen de fondo, realizado con fundamento en el articulo 45 de la Decision 486 de la Comisión de la Comunidad Andina, se le comunica

1. Documentos estudiados

- Para el presente estudio se tuvo en cuenta el capitulo descriptivo que se encuentra en los folios 2 a 15, los dibujos que se encuentran en los folios 21 a 22 y el capitulo reivindicatorio que se encuentra en los folios 60 a 63 que comprende once (11) reivindicaciones
- De acuerdo con el folio 52 de la solicitud en estudio, se considerara la prioridad reivindicada sobre la solicitud de Suecia No 9903070-2 del 30 de agosto de 1999

2. Objeto de la invención

- La invención propuesta se refiere a una estructura absorbente en un articulo absorbente, que comprende un material de espuma comprimida que se expande al humedecerse La invención se relaciona además con un metodo de produccion de la estructura absorbente y un artículo absorbente que contiene la estructura absorbente de acuerdo con la invencion

En la **reivindicación independiente 1** se define una estructura absorbente en un articulo absorbente tal como un pañal, pañal pantalón, guarda de incontinencia, toalla sanitaria, cubierta de herida, proteccion de camas, etc Y que comprende un material de espuma comprimida (1) que se expande al humedecerse, **caracterizado porque** el material (1) comprende al menos dos capas integradas (2, 3, 4) que tienen diferentes tamaños de poro medio

En la **reivindicación independiente 7** se define un metodo de producción de una estructura absorbente en un articulo absorbente tal como un pañal, guarda de incontinencia, toalla sanitaria, cubierta de herida, proteccion de camas, etc , **caracterizado en**

Formar separadamente al menos dos materiales de espuma diferente que tienen diferentes tamaños de poro medio,

Aplicar los materiales de espuma sobre la parte superior de cada uno mientras que no esté seco

Secar y comprimir las capas del material combinado de espuma

En la **reivindicación independiente 11** se define un artículo absorbente tal como un pañal, pañal pantalón, una guarda de incontinencia, una toalla sanitaria, una cubierta de herida, una protección de camas, etc. De la clase que contiene una lámina superior permeable a los líquidos (5), una lámina posterior impermeable a los líquidos (6) y una estructura absorbente aplicada entre ellos, **caracterizado porque** esta contiene una estructura absorbente (1) como se reivindica en cualquiera de las reivindicaciones 1 a 6

- Se busca mejorar la función de una estructura absorbente en la forma de un material de espuma comprimida especialmente con relación a la capacidad de adquisición y distribución. Existe referencia adicional a un método de producción de una estructura absorbente
- El objeto de la invención definido se cataloga como **A61L 15/60** y **A61F 13 15**, según la Clasificación Internacional de Patentes (IPC)

3. Determinación del estado de la técnica

El artículo 16 de la Decisión 486 de la Comisión establece "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

Solo 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 este incluido en la solicitud de fecha anterior cuando ella se publique o hubiese transcurrido el plazo previsto en el artículo 40 "

- Se consultaron las bases de datos con que cuenta y tiene acceso la entidad y se consideró el **30 de agosto de 1999** como fecha para investigar el estado de la técnica
- De acuerdo con lo establecido en el artículo 16 se determinó el estado de la técnica y se encontraron los siguientes antecedentes

No	Documento No	Título	Fecha de Publicación
1	WO9621680	"ABSORBENT FOAM MATERIALS FOR AQUEOUS FLUIDS MADE FROM HIGH INTERNAL PHASE EMULSIONS HAVING VERY HIGH WATER TO OIL RATIOS"	18 de julio de 1996
2	US5869171	"HETEROGENEOUS FOAM MATERIALS"	9 de febrero de 1999

4. Evaluación de los requisitos de patentabilidad

El artículo 14 de la Decisión 486 de la Comisión de la Comunidad Andina establece "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 con este artículo, se procedió a evaluar los requisitos de novedad, nivel inventivo y aplicación industrial con el fin de establecer la patentabilidad de la invención reivindicada en la solicitud en estudio

Evaluación del Nivel Inventivo.

El art 18 de la Decisión 486 de la Comisión de la Comunidad Andina establece "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 "

Respecto al estado de la técnica

- En el documento **WO9621680** (llamado de aquí en adelante D1) se divulgan **materiales absorbentes colapsados de baja densidad** que al contacto con fluidos acuosos, particularmente orina, pueden expandirse y absorber tales fluidos. Estas espumas de baja densidad tienen un espesor expandido desde 6 a 10 veces el espesor de la espuma en estado colapsado. Estas espumas de baja densidad son elaboradas por polimerización de emulsiones de fase interna elevada (HIPE) donde la razón de volumen a peso de la fase acuosa a la fase aceitosa está en el rango de desde alrededor de 55:1 a 100:1.
- En el documento **US5869171** (llamado de aquí en adelante D2) se divulga una estructura de espuma polimérica (HIPE) de celdas abiertas interconectadas, donde la estructura de espuma tiene por lo menos dos regiones diferentes. Tales espumas heterogéneas tienen varias aplicaciones, tales como absorción de fluidos, aislante y filtrado.

La invención además se relaciona con espumas poliméricas absorbentes que en contacto con fluidos acuosos del cuerpo pueden adquirir, distribuir y almacenar dichos fluidos.

Estas espumas de la invención tienen por lo menos dos regiones distintas de diferente densidad, composición polimérica, propiedades de superficie y/o morfología microcelular.

Esta invención además se relaciona al proceso para obtener las espumas heterogéneas por polimerización de una emulsión de agua en aceite de elevada fase interna. Y en un aspecto, el proceso utiliza por lo menos dos distintos HIPE, con cada emulsión teniendo una relativa pequeña cantidad de una fase oleosa y una gran cantidad de fase acuosa.

Respecto a la solicitud en estudio

- El técnico de nivel medio versado en la materia encargado de resolver el problema técnico puede ser un ingeniero químico que labore en la industria de los artículos absorbentes y que atienda a planes de mejoramiento continuo normalmente formulados por las empresas.

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- El principio técnico sobre el cual se fundamenta la invención propuesta es la incorporación de una estructura absorbente que presente dos diferentes tipos de poros en ella misma. Esta característica técnica ya ha sido lograda según se divulga en D2 y de hecho se logra desde el proceso de producción ya que se emplean dos fases acuosas internas de diferente corte y tamaño de glóbulos que al polimerizar resultan en diferentes tamaños de poro de la espuma.

Dado lo anterior, es posible afirmar que un técnico de nivel medio versado en la materia que conoce la importancia de mejorar las propiedades de adquisición, distribución y almacenamiento, podría desarrollar una estructura absorbente con diferentes tamaños de poro medio teniendo acceso a las suficientes enseñanzas contenidas en D2. Incluso si tiene en cuenta las enseñanzas de D1, se vería motivado a reducir en lo posible el diámetro haciendo la estructura absorbente colapsable.

Así las cosas, si el técnico de nivel medio toma las enseñanzas tanto de D1 como de D2 podría realizar productos equivalentes a los propuestos con la presente solicitud. Entonces, no podría afirmarse que exista un efecto sorprendente ni un salto tecnológico sobre lo convencional.

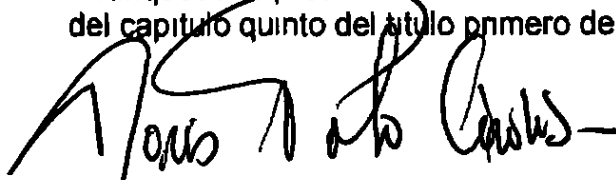
- Por lo anteriormente expuesto, la invención definida por las reivindicaciones 1 a 6 y 11 de la solicitud, podría verse afectada en su nivel inventivo ya que se deriva de manera evidente del estado de la técnica y puede ser obvia para un técnico de nivel medio versado en la materia. Por tanto, no cumpliría con las disposiciones del artículo 18 de la Decisión 486 de la Comisión de la Comunidad Andina.
- En conclusión los requisitos de patentabilidad de la presente solicitud se encuentran afectados así:

No.	Documento	Reivindicaciones Afectadas	Requisito que afecta
1	WO9621680	1 – 6, 11	Nivel Inventivo
2	US5869171		

Se anexan los apartes principales de las anterioridades en el documento adjunto.

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 No. 10 de 2001.



DORIS ELENA POLO CORDOBA (E)
Jefe de División de Nuevas Creaciones

19 MAYO 2005

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

CONCEPTO TECNICO No 672	EXAMINADOR TECNICO Código No 202042
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109 MAY 10 2008



US005869171A

United States Patent [19]
Shiveley et al.

[11] Patent Number. 5,869,171
[45] Date of Patent: Feb 9, 1999

- [54] HETEROGENEOUS FOAM MATERIALS
- [75] Inventors Thomas Michael Shiveley Moscow,
Thomas Allen DesMarais John
Collins Dyer both of Cincinnati Keith
Joseph Stone, Fairfield all of Ohio
- [73] Assignee The Procter & Gamble Company,
Cincinnati, Ohio

- [21] Appl No 87,169
[22] Filed May 29, 1998

Related U.S. Application Data

- [62] Division of Ser No 612,643 Mar 8 1996
- [51] Int. Cl.⁴ H32H 3/26
- [52] U.S. Cl. 428/304.4, 521/62, 521/63,
521/64, 521/65 521/70 604/358 604/369
- [58] Field of Search 428/304.4, 604/358,
604/369, 521/64 63 62, 65, 70

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5,268,221	12/1993	DesMarais et al	428/286
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(List continued on next page.)

Primary Examiner—Morton Locak

Attorney, Agent or Firm—Carl J Root, L Kelly Linman
Jacobus C Rasser

[57] ABSTRACT

The present invention relates to a HIPL-derived heterogeneous polymeric foam structure of interconnected open-cells wherein the foam structure has at least two distinct regions. Such heterogeneous foams have various applications, such as energy and fluid absorption, insulation, and filtration.

The invention further relates to a heterogeneous absorbent polymeric foam that upon contact with aqueous fluids (in particular body fluids such as urine and blood) can acquire, distribute, and store these fluids.

The foams of the invention have at least two distinct regions having different density, polymer composition, surface properties, and/or microcellular morphology.

The invention further relates to a process for obtaining the heterogeneous foams by polymerizing a high internal phase water-in-oil emulsion or HIPT. In one aspect the process utilizes at least two distinct HIPLs with each emulsion having a relatively small amount of an oil phase and a relatively greater amount of a water phase.

18 Claims, 13 Drawing Sheets

HETEROGENEOUS FOAM MATERIALS

This is a divisional application of Ser. No. 08/612,643 filed Mar. 8, 1996.

FIELD OF THE INVENTION

This application relates to flexible, microporous, open-celled polymeric foam materials having two or more distinct regions of different types of foam interspersed so as to deliver a desired mixture of properties in a single piece of material.

BACKGROUND OF THE INVENTION

Flexible open-celled polymeric foams are widely used for energy absorption or insulation (thermal, acoustic, mechanical), filtration, absorption of fluids and the like. In most cases, the foams desired for these purposes have relatively homogeneous structures comprising cells within a given size range joined by open windows or holes to adjacent cells. Among the various measures of such foams that are important for each application are cell size/hole size and distribution, anisotropy, proportion of cell struts to windows, and porosity. Anisotropy can arise from the relative deviation between the shape of all the cells in the foam and some geometric ideal. In general, work has been devoted to making foams as homogeneous and isotropic as possible with respect to cell size and shape and density.

Polyurethane foams having a range of densities are known materials. For example, the integral skin flexible polyurethane foams have high density skin layers that transition gradually over 1-3 cm into a lower density core region. See, for example, Ashida, K., Iwasaki, K. In *Handbook of Plastic Foams*, Lindrock, A. H., Ed., Noyes, 1995, Chapter 2, pp. 56-64-67 incorporated herein by reference. The overall densities of such foams are typically between about 200 and 1,100 kg/m³. These foams do not exhibit distinct regions having different compositional or microstructural properties within a single piece. Typically these foams also have higher densities, larger cell sizes and/or larger hole sizes than foams which may be preferred for certain applications.

Foams may be made from polymer networks which have been entangled to form an interpenetrating network (IPN). IPNs may exhibit some of the properties of both polymer types. See, for example, Odian, G. G. *Principles of Polymerization*, 3rd edition, Wiley-Interscience, New York, 1991, New York, pp. 149-150. IPNs do not inherently relate to control over any features at a supramolecular scale (e.g., density or cell size).

Laminates or sandwiches of two or more layers of foams having differing properties are also well known. See, for example, Gibson, L. J., Ashby, M. F. *Cellular Solids*, Pergamon Press, Oxford, 1988, Chapter 9. Formation of such composites requires an additional step and may require use of adhesive which may interfere with the functioning or weight of the foam composite and serves as a potential point of failure.

The development of highly absorbent articles for use as disposable diapers, adult incontinence pads and briefs and sanitary napkins, is the subject of substantial commercial interest. The ability of such products to acquire, distribute, and store fluids such as are found in body exudates (e.g., urine, sweat, feces and menses) is obviously critical to their function. Historically this has been primarily achieved by using a combination of cellulosic fibers and interspersed superabsorbent particles (generally lightly crosslinked partially neutralized polyacrylic acid that forms a gel when

exposed to free water). This approach has, however, encountered a number of difficulties in achieving efficient removal of fluid from the body of the wearer and storage away from the wearer in part due to the difficulty in controlling and maintaining the appropriate blend of particulate and fiber to provide the desired degree of capillary fluid transport and core integrity and flexibility.

Other absorbent materials capable of providing capillary fluid transport include certain types of polymeric foams in absorbent articles for the purpose of imbibing wicking and/or retaining aqueous body fluids. See, for example, U.S. Pat. No. 3,563,243 (Lindquist), issued Feb. 6, 1971 (absorbent pad for diapers and the like where the primary absorbent is a hydrophilic polyurethane foam sheet); U.S. Pat. No. 4,554,297 (Dabi) issued Nov. 19, 1985 (body fluid absorbing cellular polymers that can be used in diapers or catamenial products); U.S. Pat. No. 4,740,520 (Garvey et al.), issued Apr. 26, 1988 (absorbent composite structure, such as diapers, feminine care products and the like that contain sponge absorbents made from certain types of super-wicking, crosslinked polyurethane foams). These foams can provide core integrity and flexibility but not the desired degree of capillary fluid transport.

The use of appropriate absorbent foams in absorbent articles such as diapers and catamenial pads can provide features of capillary fluid acquisition, transport and storage required for use in high performance absorbent cores. Absorbent articles containing such foams can possess desirable wet integrity, can provide suitable fit throughout the entire period the article is worn, and can minimize changes in shape during use (e.g., swelling, bunching).

Particularly suitable absorbent low density open-celled foams have been made from High Internal Phase Emulsions (hereafter referred to as "HIPEs"). See, for example, U.S. Pat. No. 5,260,345 (DesMarais et al.), issued Nov. 9, 1993 and U.S. Pat. No. 5,268,224 (DesMarais et al.) issued Dec. 7, 1993. Other suitable absorbent foams are described in co-pending applications U.S. Ser. No. 08/370,922 (DesMarais et al. filed Jan. 10, 1995), U.S. Ser. No. 08/370,695 (Stone et al., filed Jan. 10, 1995), U.S. Ser. No. 08/370,697 (Dyer, filed Jan. 10, 1995), and U.S. Ser. No. 08/520,793 (DesMarais filed Aug. 30, 1995), all incorporated by reference herein. The HIPE process provides facile control over the cell and hole size and distribution, proportion of cell struts to windows, and porosity in these foams. When suitably treated to render the surface of these foams hydrophilic, these HIPE foams provide desirable fluid handling properties including (a) relatively good wicking and fluid distribution characteristics to transport the imbibed urine or other body fluid away from the initial impingement zone and into the unused balance of the foam structure to allow for subsequent gushes of fluid to be accommodated, and (b) a relatively high storage capacity with a relatively high fluid capacity under load, i.e., under compressive forces. These HIPE absorbent foams are also sufficiently flexible and soft so as to provide a high degree of comfort to the wearer of the absorbent article, and can be made relatively thin until subsequently wetted by the absorbed body fluid.

An important issue associated with the fluid handling properties of an absorbent foam is capillary structure. Foams having larger cell sizes and hole sizes tend to acquire fluid quickly but do not distribute fluid sufficiently against the force of gravity, nor do they store fluid effectively. Conversely, foams having smaller cell sizes and hole sizes are able to wick fluid against the force of gravity and store the fluid tightly to keep it away from the skin of the wearer.

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but are typically slower to acquire fluid. As indicated, heretofore, these opposing functions in an absorbent article have been achieved primarily by layering in the z-direction, different types of distinct absorbent foams which provide different functions. This adds complexity and cost to the process and can limit the product designs desired to those which allow for combination of individual pieces. Also, these designs are limited in being able to move fluids by differential capillary pressure only between the layers in the z-dimension of the articles, not in the x-y dimension or within the plane of a given layer.

Another important issue is the strength or resistance to compression deflection of the foam. Foams having comparatively higher densities, higher Tgs (defined hereinafter) and/or higher crosslinker levels generally exhibit greater resistance to deforming under pressure. This is achieved at the expense of using more polymer per unit volume, having a foam which deforms too slowly for practical use, or a foam which is too brittle or inflexible.

Accordingly, it would be desirable to be able to make an open-celled polymeric foam material that combines the various properties listed above as distinct regions within one material that (1) expresses a combination of cell sizes and/or hole sizes in different distinct regions of a single piece of foam, and/or (2) expresses a combination of strengths and flexibilities in a single piece of foam and/or (3) expresses a combination of energy absorbent properties in a single piece of foam. This can circumvent, at least partially, the need to use separate pieces of foams to serve different purposes, thereby enhancing the efficiency and simplicity of articles made with the objects of the current invention.

SUMMARY OF THE INVENTION

The present invention relates to a HIPL-derived heterogeneous polymeric foam structure of interconnected open-cells wherein the foam structure has at least two distinct regions. Such heterogeneous foams have various applications such as energy and fluid absorption, insulation and filtration.

The invention further relates to a heterogeneous absorbent polymeric foam that upon contact with aqueous fluids (in particular body fluids such as urine and blood), can acquire, distribute, and store these fluids. Preferably, the heterogeneous absorbent polymeric foam of the present invention comprises a hydrophilic, flexible, nonionic polymeric foam structure of interconnected open-cells.

Regardless of whether the heterogeneous foam of the present invention is relatively hydrophobic, or is a hydrophilic foam suitable for aqueous fluid absorption, the foam has at least two distinct regions that differ with regard to one or more of foam density, polymer composition, specific surface area, or microcellular morphology (e.g., cell size, shape, or distribution, or hole size).

The present invention further relates to a process for obtaining the heterogeneous foams by polymerizing a water-in-oil emulsions or HIPE. In one aspect, the process utilizes at least two distinct HIPEs, with each emulsion having a relatively small amount of an oil phase and a relatively greater amount of a water phase. This process comprises the steps of:

A) forming a first water-in-oil emulsion from

1) an oil phase comprising:

a) from about 85 to about 98% by weight of a monomer component capable of forming a copolymer having a Tg of about 95° C or lower, the monomer component comprising:

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i) from about 20 to about 70% by weight of at least one substantially water-insoluble monofunctional monomer capable of forming an atactic amorphous polymer having a Tg of about 25° C or lower;

ii) from about 10 to about 50% by weight of at least one substantially water-insoluble monofunctional comonomer capable of imparting toughness about equivalent to that provided by styrene;

iii) from about 5 to about 50% by weight of a first substantially water-insoluble, polyfunctional crosslinking agent selected from divinylbenzenes, trivinylbenzenes, divinyltoluenes, divinylxylenes, divinylphthalenes, divinylalkylbenzenes, divinylphenanthrenes, divinylbiphenyls, divinylidiphenylmethanes, divinylbenzyls, divinylphenylethers, divinylidiphenylsulfides, divinylfurans, divinylsulfide, divinylsulfone, and mixtures thereof; and

iv) from 1 to about 15% by weight of a second substantially water-insoluble polyfunctional crosslinking agent selected from polyfunctional acrylates, methacrylates, acrylamides, methacrylamides and mixtures thereof; and

b) from about 2 to about 15% by weight of an emulsifier component which is soluble in the oil phase and which is capable of forming a stable water-in-oil emulsion, the emulsifier component comprising a primary emulsifier having at least about 40% by weight emulsifying components selected from diglycerol monoesters of branched C₁₆-C₂₄ fatty acids, linear unsaturated C₁₆-C₂₂ fatty acids, and linear saturated C₁₂-C₁₄ fatty acids; sorbitan monoesters of branched C₁₆-C₂₁ fatty acids, linear unsaturated C₁₆-C₂₂ fatty acids, linear saturated C₁₂-C₁₁ fatty acids, diglycerol monoaliphatic ethers of branched C₁₆-C₂₁ alcohols, linear unsaturated C₁₆-C₂₂ alcohols, and linear saturated C₁₂-C₁₁ alcohols, and mixtures thereof; and

2) a water phase comprising an aqueous solution containing (a) from about 0.2 to about 20% by weight of a water-soluble electrolyte and (b) an effective amount of a polymerization initiator;

3) a volume to weight ratio of water phase to oil phase in the range of from about 20:1 to about 200:1;

B) optionally forming a second distinct water-in-oil emulsion which is different in at least one respect from the first emulsion but which comprises materials and ranges selected from those listed in parts A)1), A)2) and A)3), and

C) either

1) a) combining the first and second water-in-oil emulsions in a forming vessel prior to polymerizing the monomer components of either of the water-in-oil emulsions; and

b) polymerizing both emulsions to form a polymeric, heterogeneous foam;

2) a) partially or completely polymerizing the monomer components in the oil phase of the first water-in-oil emulsion;

b) combining the material from step C)2a) and the second water-in-oil emulsion; and

c) polymerizing the second emulsion and the first emulsion if combined when partly cured to provide a polymeric, heterogeneous foam; or

3) making only the first water-in-oil emulsion while varying the process conditions by which it is formed in a regular fashion using a single emulsion forming head as described hereinafter

In one embodiment one of the emulsions generated may be poured into a container or reservoir partially or completely filled with spherical segments or strips of material prepared from a previously polymerized second emulsion. These spherical segments or strips of material are thoroughly saturated with an aqueous solution closely approximating the composition of the aqueous phase used to form the emulsion added. This mixture is then cured at an appropriate temperature to make a heterogeneous foam of the present invention.

In another embodiment, striped segments of unlike material may be prepared using two or more emulsion forming nozzles expressing different emulsions, layer upon layer in a forming vessel, or by rhythmically varying the emulsion making conditions with the single emulsion nozzle. In this case, control over the relative rates of pouring and the relative dispositions of the two nozzles provides control over the spatial relationships of the two (or more) different types of polymeric material developed in the final cured foam.

In a third embodiment a single emulsion is produced but one or more of the conditions of shear, water-to-oil ratio, pour temperature, and the like are rhythmically or continuously varied so as to produce regions in the resultant foam of different properties. Thus, unlike the two embodiments described above, formation of the optional second emulsion is not necessary in this embodiment.

In a fourth embodiment a first unpolymerized emulsion is introduced into a form containing a second emulsion which is not substantially polymerized. By introducing the first emulsion from a nozzle positioned at the bottom of the form while withdrawing the nozzle, the emulsion forms a column or other geometric figure within the second emulsion. The process is repeated. Slices of the cured emulsion thus possess regularly repeating cross sections of the first emulsion in the form of the geometric figure imbedded in the plane of the slice of the second emulsion. Thus, regions of differing properties are introduced within a sheet of material.

In a fifth embodiment a first emulsion (uncured or cured) is molded or cut to a shape that acts as a mold or fits into a mold for an additional amount of a distinct emulsion which provides irregular, non-planar cross sections of intimately contacted material in the final cured product.

In a sixth embodiment, a single emulsion is subjected to centrifugation prior to polymerization. Conditions of the centrifugation are mild enough that the water droplets are not ruptured to disrupt the emulsion, but stringent enough to form a density gradient within the emulsion. Upon curing, a density gradient will exist in the foam.

Of course the skilled artisan will recognize that more than two distinct emulsions can be employed so as to provide a heterogeneous foam comprising regions with more than two different properties.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG 1 of the drawings is a photomicrograph (250x magnification) of the first region of a heterogeneous foam having four regions and three interfaces.

FIG 2 of the drawings is a photomicrograph (250x magnification) of the second region within the same heterogeneous foam.

FIG 3 of the drawings is a photomicrograph (250x magnification) of the second interface of the same heterogeneous foam where individual pour lines intersect between the second and third regions.

FIG 4 of the drawings is a photomicrograph (250x magnification) of the third region within the same heterogeneous foam.

FIG 5 of the drawings is a photomicrograph (250x magnification) of the third interface of the same heterogeneous foam where individual pour lines intersect between the third and fourth regions.

FIG 6 of the drawings is a photomicrograph (250x magnification) of the fourth region within the same heterogeneous foam.

FIG 7 of the drawings is a photomicrograph (30x) of the first of the three interfaces within the same heterogeneous foam.

FIG 8 of the drawings is a photomicrograph (30x) of the second of the three interfaces within the same heterogeneous foams.

FIG 9 of the drawings is a photomicrograph (30x) of the third of the three interfaces within the same heterogeneous foams.

FIG 10 is a top plan view of a catamenial product having a heterogeneous foam of the present invention as an absorbent member.

FIG 11 is a cutaway depiction of a disposable diaper that utilizes the absorbent polymeric foam of the present invention as an hourglass-shaped fluid storage/distribution component optionally in an absorbent diaper core of dual-layer configuration.

FIG 12 of the drawings represents a cut-away view of a form-fitting article such as a disposable training pants product that employs an absorbent polymeric foam according to the present invention as an absorbent core.

FIG 13 of the drawings is a perspective view of a single piece of heterogeneous foam of the present invention having three distinct regions where the middle region is a relatively large celled structure and the two outer regions are both relatively small celled structures. Alternatively the distinct regions may differ with regard to capillary suction specific surface area per volume.

FIG 14 of the drawings is a perspective view of a single piece of heterogeneous foam of the present invention having 5 distinct regions.

FIG 15 of the drawings is a perspective view of the same foam structure as that depicted in FIG 13, where the foam is folded so as to provide a structure having a relatively large celled structure on the top and a relatively small celled structure on the bottom. This foam structure is particularly suited for use as an absorbent article such as a catamenial pad and provides both an acquisition/distribution layer and a storage layer in one piece of foam.

FIG 16 is a perspective view of a single piece of heterogeneous foam having discontinuous spheres located within a discrete continuous region.

FIG 17 is a cross-sectional view of the side of a single piece of heterogeneous foam of the present invention having discrete regions in the z direction (i.e. thickness) of the foam.

FIG 18 is a perspective view of the same piece of heterogeneous foam shown in FIG 17 again showing the discrete regions in the z-direction of the foam.

FIG 19 is a photomicrograph (250x magnification) of a heterogeneous foam having discrete unconnected regions dispersed in a continuous region. The photomicrograph is believed to be taken at the interface of the continuous and at least one dispersed region. Surprisingly, no discontinuity is observed between the regions.

sition of body fluid. Outer regions 52 and 56 will preferably comprise relatively small celled material, again to prevent leakage of fluid from the sides of the corresponding product. Regions 53 and 55 will both preferably comprise material that have cell sizes smaller than region 54 but larger than regions 52 and 56. As a core for a feminine hygiene pad outer regions 52 and 56 may have hole sizes smaller than 8 μ m to preclude passage of red blood cells into the outer edges of the product which might signal impending side-leakage and failure to the consumer. Such regions will absorb free fluid from the menses without passing red blood cells and attendant color.

The foam depicted in FIGS. 17 and 18 is illustrative of an embodiment where the distinct regions are layered in the z direction. FIG. 17 is a side view of foam 70 having upper region 72 and lower region 74. FIG. 18 is a perspective view, again showing upper region 72 and lower region 74. This type of foam is particularly suitable as an absorbent material, such as a diaper wherein upper region 72 is an acquisition material, and lower region 74 is a storage material. A foam having regions layered in the z-direction can be formed by pouring an emulsion to form region 74 in a mold and simultaneously or sequentially adding a second emulsion to form region 72. In a preferred embodiment, region 72 will have a relatively lower specific surface area per volume than region 74, which will allow region 74 to drain fluid from region 72.

IV Test Methods

Many of the test methods used require a larger sample of homogeneous material than may be present in the heterogeneous foams of the present invention. Measurement of the properties of the distinct regions must then be obtained by taking a sample of the distinct emulsion streams, curing them identically and using them to determine the specific independent properties of the heterogeneous regions of the foam.

A Dynamic Mechanical Analysis (DMA)

DMA is used to determine the T_gs of polymers including polymeric foams. Samples of the foams are sliced into blocks 3–5 mm in thickness and washed 3–4 times in distilled water expressing the fluid through roller nips between each washing. The resulting foam blocks are allowed to dry in air. The dried foam slices are cored to yield cylinders 25 mm in diameter. These cylinders are analyzed using a Rheometrics RSA II dynamic mechanical analyzer set in compression mode using parallel plates 25 mm in diameter. Instrument parameters used were as follows:

Temperature: step from ca. 85° C to –40° C in steps of 25° C.

Soak intervals between temperature changes: of 125–160 seconds.

Dynamic strain: set at 0.1% to 1.0% (usually 0.7%).

Frequency: set at 1.0 radians/second.

Auto tension: set in static force tracking dynamic force mode with initial static force set at 5 g.

The glass transition temperature is taken as the maximum point of the loss tangent versus temperature curve. It is appropriately measured only on a homogeneous portion of the heterogeneous foam, or from a representative piece of homogeneous foam prepared under the same conditions as the region from the heterogeneous foam.

B Resistance to Compression Deflection (RTCD)

Resistance to compression deflection can be quantified by measuring the amount of strain (% reduction in thickness) produced in a foam sample which has been saturated and expanded with synthetic urine after a confining pressure of

0.74 psi (5.1 kPa) has been applied to the sample as described in the TEST METHODS section of U.S. Pat. No. 5,387,207 (Dyer et al.) issued Feb. 7, 1995. Resistance to Compression Deflection measurements are typically made on the same sample concurrently with the measurement of Free Absorbent Capacity and Expansion Factor as described below.

C Free Absorbent Capacity

Free absorbent capacity can be quantified by measuring the amount of synthetic urine absorbed in a foam sample which has been saturated and expanded with synthetic urine. Free Absorbent Capacity measurements are typically made on the same sample concurrently with the measurement of Resistance to Compression Deflection and Expansion Factor as described in the TEST METHODS section of U.S. Pat. No. 5,387,207 (Dyer et al.) issued Feb. 7, 1995.

D Interfacial tension (IFT) method (Spinning Drop)

Interfacial tension (IFT) is measured at 50° C. by the spinning drop method described in U.S. Pat. No. 5,387,207 (Dyer et al.) issued Feb. 7, 1995 (herein incorporated by reference) except that (1) the monomer mixture used in preparing the oil phase contains styrene, divinylbenzene (55% technical grade), 2-ethylhexylacrylate, and 1,4-butanediol dimethacrylate in a weight ratio of 14:14:60:12; (2) the concentration of emulsifier in the oil phase is varied by dilution from an upper concentration of generally about 5–10 weight % down to a concentration where the IFT increases to a value that is at least about 10 dyne/cm greater than the minimum IFT, or about 18 dyne/cm whichever is less; (3) a smooth line drawn through a plot of IFT versus log emulsifier concentration is used to determine the minimum IFT; (4) the Critical Aggregation Concentration (CAC) is determined by extrapolating the low-concentration generally linear portion of the IFT versus log concentration plot (i.e., the portion of the curve typically used to calculate surface area per molecule at the interface, see for example, Rosen, M. J., *Surfactants and Interfacial Phenomena*, 2nd ed. Wiley & Sons, New York, 1989, pp. 64–69) to higher concentration; the emulsifier concentration on this extrapolated line corresponding to the minimum IFT is taken as the CAC. Generally an upper emulsifier concentration of about 5–10 weight % is used. Desirably, the upper emulsifier concentration used is at least about twice (more desirably at least about three times) the CAC of the emulsifier. For emulsifiers having a solubility in the oil phase at ambient-temperature of less than 5 wt %, the upper concentration limit can be reduced as long as this concentration is still at least about twice the CAC of the emulsifier at 50° C.

E Capillary Absorption Pressures

A capillary absorption isotherm curve is generated using the Vertical Wicking Absorbent Capacity test described in the TEST METHODS section of U.S. Pat. No. 5,387,207 (Dyer et al.) issued Feb. 7, 1995, which is incorporated by reference except at 31° C. rather than 37° C. The curve is a plot of the absorbent capacity of each segment as a function of wick height, using the distance from the top of the water reservoir to the midpoint of each segment for the height h. The capillary absorption pressure is taken as the height of the foam that has an absorbent capacity one-half of the foam's free absorbent capacity.

V Specific Examples

These examples illustrate the specific preparation of foams from HIPEs according to the present invention.

EXAMPLE 1

Preparation of a Heterogeneous Foam from Two HIPE Streams HIPE Preparation

76 83
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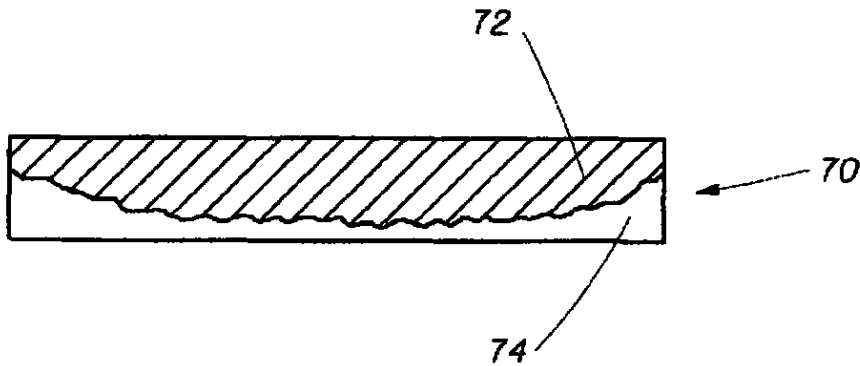


Fig 17

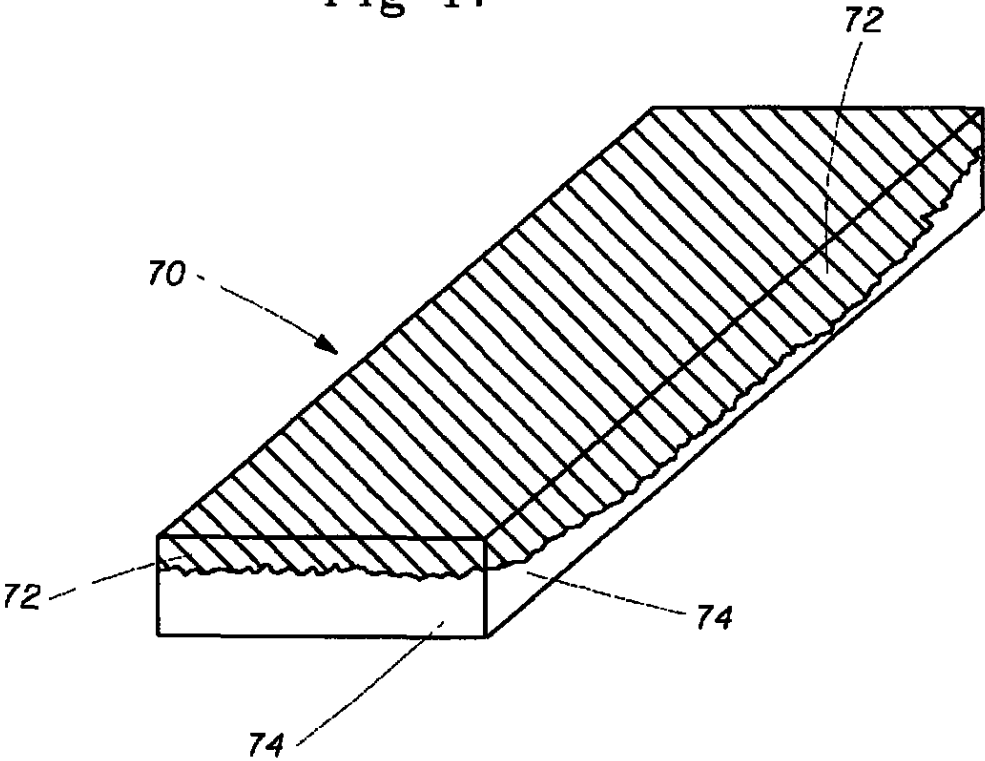


Fig 18

PCT

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(21) International Application Number PCT/US96/00084 (22) International Filing Date 11 January 1996 (11.01 96) (30) Priority Data 08/370,922 10 January 1995 (10 01 95) US (71) Applicant THE PROCTER & GAMBLE COMPANY [US/US], One Procter & Gamble Plaza Cincinnati OH 45202 (US) (72) Inventors DesMARAIS, Thomas, Allen, 10204 Scull Road Cincinnati OH 45252 (US) STONE Keith Joseph 1923 August Boulevard, Fairfield, OH 45014 (US) DYER, John, Collins, 3760 Sherbrooke Drive Cincinnati, OH 45241 (US) HIRD, Bryn, 8519 Eagle Creek, Cincinnati, OH 45224 (US) GOLDMAN, Stephen Allen, 1260 Forest Court, Cincinnati, OH 45215 (US) PEACE, Michelle Renee 206 East Main Street #2, Mason, OH 45040 (US) SEIDEN Paul, 2890 Grandin Road Cincinnati OH 45208 (US) (74) Agents REED, T, David et al The Procter & Gamble Company, 5299 Spring Grove Avenue, Cincinnati, OH 45217 (US)		(81) Designated States AL AM, AT AU, AZ, BB, BG BR BY, CA, CH, CN, CZ, DE DK, EE, ES, FI, GB GE, HU IS JP, KE, KG KP, KR KZ, LK, LR LS LT, LU LV MD MG MK, MN, MW VX, NO NZ, PL, PT, RO, RU, SD SE, SG, SI SK, TJ TM TR, TT UA UG UZ, VN, ARIPO patent (KE LS MW SD SZ, UG), Eurasian patent (AZ, BY KZ, RU TJ TM) European patent (AT BE, CH DE, DK ES, FR GB, GR, IE IT LU MC NL PT, SE), OAPI patent (BF, BJ CF CG, CI, CM, GA, GN, ML, MR NE SN TD TG) Published <i>With international search report</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments</i>
(54) Title ABSORBENT FOAM MATERIALS FOR AQUEOUS FLUIDS MADE FROM HIGH INTERNAL PHASE EMULSIONS HAVING VERY HIGH WATER-TO-OIL RATIOS (57) Abstract Low density collapsed absorbent foams materials that, upon contact with aqueous fluids, in particular urine, can expand and absorb these fluids. These low density foams typically have an expanded thickness from about 6 to about 10 times the thickness of the foams in their collapsed state. These low density foams are made by polymerizing high internal phase emulsions (HIPEs) where the volume to weight ratio of the water phase to the oil phase is in the range of from about 55:1 to about 100:1.		

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DETAILED DESCRIPTION OF THE INVENTION

I Low Density Polymeric Foam

A General Foam Characteristics

Polymeric foams according to the present invention useful in absorbent articles and structures are those which are relatively open-celled. This means the individual cells of the foam are in complete, unobstructed communication with adjoining cells. The cells in such substantially open-celled foam structures have intercellular openings or "windows" that are large enough to permit ready fluid transfer from one cell to the other within the foam structure.

These substantially open-celled foam structures will generally have a reticulated character with the individual cells being defined by a plurality of mutually connected, three dimensionally branched webs. The strands of polymeric material making up these branched webs can be referred to as "struts." Open-celled foams having a typical strut-type structure are shown by way of example in the photomicrographs of Figures 1 and 2. For purposes of the present invention, a foam material is "open-celled" if at least 80% of the cells in the foam structure that are at least 1 μm in size are in fluid communication with at least one adjacent cell.

In addition to being open-celled, these polymeric foams are sufficiently hydrophilic to permit the foam to absorb aqueous fluids in the amounts specified hereafter. The internal surfaces of the foam structures are rendered hydrophilic by residual hydrophilizing surfactants left in the foam structure after polymerization, or by selected post-polymerization foam treatment procedures, as described hereafter.

The extent to which these polymeric foams are "hydrophilic" can be quantified by the "adhesion tension" value exhibited when in contact with an absorbable test liquid. The adhesion tension exhibited by these foams can be determined experimentally using a procedure where weight uptake of a test liquid, e.g., synthetic urine, is measured for a sample of known dimensions and capillary suction specific surface area. Such a procedure is described in greater detail in the TEST METHODS section of copending U.S. application Serial No. 989,270 (Dyer et al), filed December 11, 1992, which is incorporated by reference. Foams which are useful as absorbents in the present invention are generally those which exhibit an adhesion tension value of from about 15 to about 65 dynes/cm, more preferably from about 20 to about 65 dynes/cm, as determined by capillary absorption of synthetic urine having a surface tension of 65 ± 5 dynes/cm.

The polymeric foams of the present invention can be prepared in the form of collapsed (i.e. unexpanded), polymeric foams that, upon contact with aqueous fluids,

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5 expand and absorb such fluids. These collapsed polymeric foams are usually obtained by expressing the water phase from the polymerized HIPE foam through compressive forces, and/or thermal drying and/or vacuum dewatering. After compression, and/or thermal drying/vacuum dewatering, the polymeric foam is in a collapsed, or, unexpanded state.

10 The cellular structure of a representative collapsed HIPE foam from which water has been expressed by compression is shown in the photomicrograph of Figures 3 and 4. As shown in these Figures, the cellular structure of the foam is distorted, especially when compared to the expanded HIPE foam structures shown in Figures 1 and 2. As can also be seen in Figures 3 and 4, the voids or pores (dark areas) in the collapsed foam structure have been flattened or elongated.

15 Following compression and/or thermal drying/vacuum dewatering, the collapsed polymeric foam can reexpand when wetted with aqueous fluids. Surprisingly, these polymeric foams remain in this collapsed, or unexpanded state, for significant periods of time, e.g., up to at least about 1 year. The ability of these
20 polymeric foams to remain in this collapsed/unexpanded state is believed to be due to the capillary forces, and in particular the capillary pressures developed within the foam structure. As used herein, "capillary pressures" refers to the pressure differential across the liquid/air interface due to the curvature of meniscus within the narrow confines of the pores in the foam. [See Chatterjee, "Absorbency," Textile Science and Technology, Vol 7, 1985, p 36.]

25 After compression, and/or thermal drying/vacuum dewatering to a practicable extent, these polymeric foams have residual water that includes both the water of hydration associated with the hydroscopic, hydrated salt incorporated therein, as well as free water absorbed within the foam. This residual water (assisted by the hydrated
30 salts) is believed to exert capillary pressures on the resulting collapsed foam structure. Collapsed polymeric foams of the present invention can have residual water contents of at least about 4%, typically from about 4 to about 40%, by weight of the foam when stored at ambient conditions of 72°F (22°C) and 50% relative humidity. Preferred collapsed polymeric foams have residual water contents of from
35 about 5 to about 25% by weight of the foam.

A key parameter of these foams is their glass transition temperature (T_g). The T_g represents the midpoint of the transition between the glassy and rubbery states of the polymer. Foams that have a higher T_g than the temperature of use can be very strong but will also be very rigid and potentially prone to fracture. Such
40 foams also typically take a long time to recover to the expanded state when wetted.