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(54) Title: USE OF SURFACTANTS TO IMPROVE AGED PROPERTIES OF FIBERGLASS INSULATION PRODUCTS

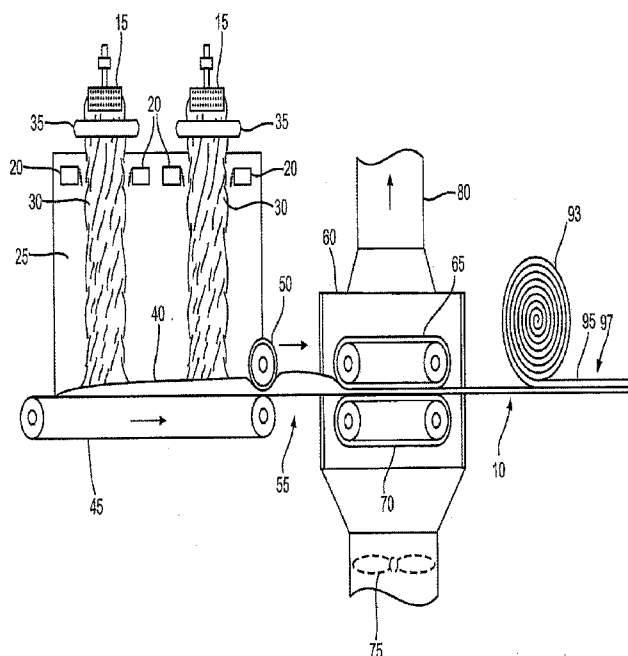


Fig. 1

(57) Abstract: Fibrous insulation products manufactured with surfactants and methods for making are disclosed. The surfactant may be neutral or charged and, if charged, may be anionic, cationic or zwitterionic, although neutral or non-ionic provide suitable results. The surfactant may be of any chemical structure class, although ethoxylated alcohols and ethoxylated ethers have been found most suitable. Surfactant may be sprayed onto mineral fibers as a separate dispersion or as part of a binder dispersion. The surfactant may optionally be used with an organo-silane coupling agent, such as an amino-silane.



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USE OF SURFACTANTS TO IMPROVE AGED PROPERTIES OF FIBERGLASS INSULATION PRODUCTS

RELATED APPLICATIONS

[0001] This application claims priority to and the benefit of U.S. Non-Provisional Patent Application Serial No. 13/495,356, filed June 13, 2012, entitled “Use of Surfactants To Improve Aged Properties of Fiberglass Insulation Products”, the entire disclosure of which is incorporated herein by reference.

BACKGROUND OF THE INVENTION

[0002] The present invention relates generally to the field of fiberglass insulation, including panels and methods to improve the product properties thereof, more specifically, mechanical properties such as sag and recovery after aging. The invention applies to all forms of fibrous insulation, including ceiling boards and tiles, wall panels, duct boards, pipe and molded or formed insulation products, but has most relevance for light to medium density fibrous insulation batts, such as used in the wall and ceiling cavities of homes and buildings.

[0003] Fibrous insulation and construction panels are typically manufactured by fiberizing a molten composition of polymer, glass or other mineral material to form fine fibers and depositing the fibers on a collecting conveyor to form a batt or a blanket. Mineral fibers, such as glass fibers, are typically used in insulation products. A binder composition may optionally be used to bond the fibers together where they contact each other. During the manufacturing process some insulation products are formed and cut to provide sizes generally dimensioned to be compatible with standard construction practices, e.g. standard sized wall or ceiling panels having widths and/or length adapted for specific building practices. Some insulation products also incorporate a facing layer or material on at least one of the major surfaces. In many cases the facing material is provided as a vapor barrier, while in other insulation products, such as binderless products, the facing material improves the product integrity. Other insulation products may be used on structures such as, for example, pipes, ducts, appliances and other devices. The use of insulation on these structures assists to

maintain a thermal difference between the structure and the environment. However, in some environments, moisture may be present and may infiltrate the insulation. This can cause the insulation to be less effective than intended and cause other issues.

[0004] Surfactants have been used in fibrous insulation products – as a component of a binder composition – primarily as a wetting agent to promote the distribution of binder throughout the fibrous product (see e.g. WO 2011/044490 to Hawkins, et al.) Hawkins et al characterize the surfactant as a process aid “to assist in binder atomization, wetting, and interfacial adhesion.” This is because natural or bio-based binders comprising starch or other polysaccharides exhibit extensive hydrogen bonding and can become very viscous and sticky, particularly at high concentrations. A surfactant or wetting agent can reduce the surface tension and more readily allow the thick binder dispersion to flow along and wet the glass fibers. In Example 11, Hawkins et al. disclose data showing a reduced surface tension in binder compositions containing various surfactants.

[0005] In Table 30, Hawkins et al. show the impact of steam aging (a form of accelerating testing) on tensile strength and tensile strength normalized by LOI content, for handsheets containing various titanate coupling agents. All coupling agent except Tyzor TPT reduced the initial ambient tensile strength/LOI ratio; and all titanate coupling agents reduced the steam-aged tensile strength/LOI ratio. Furthermore, the disparity between ambient and aged tensile strength/LOI ratios is the least when no coupling agent is used; each coupling agent tested broadened the disparity between ambient and steam-aged properties, most egregiously with Tyzor TPT.

[0006] The present invention seeks to address these problems and others.

SUMMARY OF THE INVENTION

[0007] In general, the invention relates to the addition of surfactant additives to fibrous insulation products. The addition results in certain improved properties that are unexpected; for example, improved mechanical properties of products that have been aged in hot and humid conditions.

[0008] Thus, in a first aspect, the invention provides a method for manufacturing a fibrous insulation product having improved mechanical properties upon aging, the method comprising:

forming a fibrous product from a plurality of mineral fibers,

applying a thermosetting binder to the fibers of the fibrous product, the thermosetting binder including a polyhydroxyl compound and a polycarboxylic acid, the polyhydroxyl compound and the polycarboxylic acid being capable of forming crosslinks, and

applying a surfactant to the fibers of the fibrous product, the surfactant being applied to achieve a normalized concentration of about 0.01% to about 10% based on the dry weight of the fibrous product.

[0009] In a second aspect, the invention provides a method for improving the aged mechanical properties of a fibrous insulation product, the method comprising:

forming a fibrous product from a plurality of mineral fibers and a natural, thermosetting binder, the binder including a carbohydrate-based polyhydroxyl compound and a polycarboxylic acid, the polyhydroxyl compound and the polycarboxylic acid being capable of forming crosslinks, and

applying a surfactant to the fibers of the fibrous product, the surfactant being applied to achieve a normalized concentration of about 0.05% to about 5% based on the dry weight of the fibrous product; wherein at least one aged mechanical property is improved by at least 2% compared to fibrous product not containing the surfactant

[0010] The following optional features may be provided in either aspect of the invention. The surfactant may be applied at a normalized concentration (based on the dry weight of the fibrous product) from about 0.05 % to about 1%, from about 0.01 % to about 5.0% by weight, or from about 0.05 % to about 0.5% by weight, or from about 0.1 % to about 0.5%. The surfactant may be applied as part of a binder dispersion or independently. The surfactant may be an ionic (anionic, cationic or zwitterionic) or a non-ionic surfactant. In some embodiments, the surfactant is an ethoxylated polyalcohol, such as one selected from the Surfynol® series 420, 440, 465, and 485. In some embodiments, the surfactant is applied in combination with a silane coupling agent.

[0011] The binder may be a natural binder, such as one comprising a carbohydrate-based polyhydroxyl compound and a polycarboxylic acid. The carbohydrate-based polyhydroxyl compound may comprise a polysaccharide selected from a starch, a maltodextrin, a dextrin and a syrup; or it may be a monosaccharide or disaccharide carbohydrate. In some embodiments, the carbohydrate-based polyhydroxyl compound has a dextrose equivalent (DE) from about 2 to about 20. In some embodiments, the polycarboxylic acid is selected from a polyacrylic acid or an organic di- or tri- carboxylic acid.

[0012] A feature of the invention is the surprising ability to improve a mechanical property of the fibrous insulation product. Such a mechanical property may be any one or more of: e.g. recovery, sag, compressive strength, and tensile strength. While an initial or ambient mechanical property may be affected, it is the improvement of aged properties that is most surprising. When the aged property is recovery, it may be improved by at least 2% , at least 5%, at least 10%, or more compared to the same product made with without surfactant. When the aged property is sag, it may be improved by at least 5%, at least 10%, at least 20% or more compared to the same product made with without surfactant. When the aged property is compressive strength, it may be improved by at least 5%, at least 10% or at least 15% compared to the same product made with without surfactant.

[0013] Other advantages and features and variations will become apparent to those skilled in the art from the following detailed description of various embodiments, when read in light of the accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

[0014] The accompanying drawings, incorporated herein and forming a part of the specification, illustrate the present invention in its several aspects and, together with the description, serve to explain the principles of the invention. In the drawings, the thickness of the lines, layers, and regions may be exaggerated for clarity.

[0015] Fig. 1 is a diagrammatic side elevation view of fibrous product manufacturing line; and

[0016] Figs. 2-4, are charts of data described in the examples;

DETAILED DESCRIPTION

[0017] Unless defined otherwise, all technical and scientific terms used herein have the same meaning as commonly understood by one of ordinary skill in the art to which the invention belongs. Although any methods and materials similar or equivalent to those described herein can be used in the practice or testing of the present invention, the preferred methods and materials are described herein. All references cited herein, including books, journal articles, published U.S. or foreign patent applications, issued U.S. or foreign patents, and any other references, are each incorporated by reference in their entireties, including all data, tables, figures, and text presented in the cited references.

[0018] Unless otherwise indicated, all numbers expressing ranges of magnitudes, such as angular degrees or web speeds, quantities of ingredients, properties such as molecular weight, reaction conditions, dimensions and so forth as used in the specification and claims are to be understood as being modified in all instances by the term "about." Accordingly, unless otherwise indicated, the numerical properties set forth in the specification and claims are approximations that may vary depending on the desired properties sought to be obtained in embodiments of the present invention. Notwithstanding that the numerical ranges and parameters setting forth the broad scope of the invention are approximations, the numerical values set forth in the specific examples are reported as precisely as possible. Any numerical values, however, inherently contain certain errors necessarily resulting from error found in their respective measurements. All numerical ranges are understood to include all possible incremental sub-ranges within the outer boundaries of the range. Thus, a range of 30 to 90 degrees discloses, for example, 35 to 50 degrees, 45 to 85 degrees, and 40 to 80 degrees, etc.

[0019] Fibrous insulation products are designed to block the transfer of heat. Heat may be transferred through a fibrous glass pack by three distinct methods: convection, conduction and radiation. Convection, i.e. flow of fluid (air) through the pack includes flow driven by external forces, such as wind, fans or blowers and natural or free flow driven by conditions within the pack, such as thermal or density gradients; similarly, conduction includes conduction by air, glass or any other compounds present within the pack. The term "R-value" is the commercial unit used to measure the effectiveness of thermal insulation and is the reciprocal of its thermal conductance which, for "slab" materials having substantially parallel faces, is defined as the rate of flow of thermal energy (BTU/hr or Watt) per unit area (square foot = ft^2 or square meter = m^2) per degree of temperature difference (Fahrenheit or Kelvin) across the thickness of the slab material (inches or meters). Inconsistencies in the literature sometimes confuse the intrinsic thermal properties resistivity, r , (and conductivity, k), with the total material properties resistance, R , (and conductance, C), the difference being that the intrinsic properties are defined as being per unit thickness, whereas resistance and conductance (often modified by "total") are dependent on the thickness of the material, which may or may not be 1 unit. This confusion, compounded by multiple measurement systems, produces an array of complex and confusing units the most common of which are:

	English (inch-pound)	Metric/SI units
Intrinsic resistivity, r (conductivity, k , is reciprocal)	$\frac{\text{hr} \cdot \text{ft}^2 \cdot ^\circ\text{F}}{\text{BTU} \cdot \text{in}}$	$\frac{\text{K} \cdot \text{m}}{\text{W}}$
Total material resistance, R (conductance, C , is reciprocal)	$\frac{\text{hr} \cdot \text{ft}^2 \cdot ^\circ\text{F}}{\text{BTU}}$	$\frac{\text{K} \cdot \text{m}^2}{\text{W}}$

For ease of comparisons of materials of differing thicknesses, the building industry sometimes reports thermal resistance (or conductance) per unit thickness (e.g. per inch) effectively converting it to thermal resistivity (conductivity), but retains the traditional symbol, R or R -value. It is further observed that the “conductivity” referenced above includes the total heat transfer by any of the mechanisms described above, not just by conduction. Thermal conductivity and resistivity may be measured using commercial instruments like the FOX instruments (LaserComp, Saugus, MA) according to ASTM C518 or other standard protocols.

Fibrous product manufacture

[0020] Although other types of fibrous products and manufacturing processes are known, the invention is well exemplified by the manufacture of glass fiber insulation carried out in a continuous process by rotary fiberization of molten glass as depicted in FIG. 1. Glass or other mineral material may be melted in a tank or melter (not shown) and supplied by a forehearth (not shown) to a “fiberizer” or fiber forming device such as a fiberizing spinner 15. The spinners 15 are rotated at high speeds so that centrifugal force causes the molten glass to pass through holes in the circumferential sidewalls of the fiberizing spinners 15 to form primary glass fibers. Secondary glass fibers 30 of random lengths may be attenuated from the fiberizing spinners 15 and blown generally downwardly, that is, generally perpendicular to the plane of the spinners 15, by blowers 20 positioned within a forming chamber 25. It is to be appreciated that the glass fibers 30 may all be the same type of glass or they may be formed of different types of glass, or even of other molten mineral materials. It is also within the purview of the present invention that at least one of the fibers 30 formed from the fiberizing spinners 15 is a dual glass fiber where each individual fiber is formed of two different glass compositions. The glass fibers 30 may have a diameter from about 2 to about 9 microns, or from about 3 to about 6 microns.

[0021] The blowers 20 direct the fibers 30 toward a foraminous chain or conveyor 45 to form a fibrous pack 40. The glass fibers, while in transit in the forming chamber 25 and

while still hot from the drawing operation, are sprayed with a binder composition by an annular spray ring 35 so as to result in a distribution of the binder composition throughout the formed insulation pack 40 of fibrous glass. Coolant such as water may also be applied to the glass fibers 30 in the forming chamber 25, typically by spraying using a ring system similar to ring 35. Often coolant water is applied prior to the application of the aqueous binder composition to at least partially cool the glass fibers 30. The binder is typically applied in an amount from about 1% to 30% by weight of the total fibrous product, more usually from about 2% to about 20% or from about 3% to about 17%.

[0022] The glass fibers 30 having the uncured resinous binder adhered thereto may be gathered and formed into an uncured insulation pack 40 on an endless forming conveyor 45 within the forming chamber 25 with the aid of a vacuum (not shown) drawn through the fibrous pack 40 from below the forming conveyor 45. The residual heat from the glass fibers 30 and the flow of air through the fibrous pack 40 during the forming operation are generally sufficient to volatilize a majority of the water from the binder before the glass fibers 30 exit the forming chamber 25, thereby leaving the remaining components of the binder on the fibers 30 as a viscous or semi-viscous high-solids liquid.

[0023] The coated fibrous pack 40, which is in a compressed state due to the flow of air through the pack 40 in the forming chamber 25, is then transferred out of the forming chamber 25 under exit roller 50 to a transfer zone 55 where the pack 40 vertically expands due to the resiliency of the glass fibers. The expanded insulation pack 40 is then heated, such as by conveying the pack 40 through a curing oven 60 where heated air is blown through the insulation pack 40 to evaporate any remaining water in the binder, cure the binder, and rigidly bond the fibers together. Heated air is forced through a fan 75 through the lower oven conveyor 70, the insulation pack 40, the upper oven conveyor 65, and out of the curing oven 60 through an exhaust apparatus 80. Although only one oven zone is depicted in Fig. 1, there may be multiple zones used to dry and cure the fibrous product. The cured binder imparts strength and resiliency to the insulation blanket 10, depending on the nature and properties of the particular insulation product being manufactured.

[0024] Also, in the curing oven 60, the insulation pack 40 may be compressed by upper and lower foraminous oven conveyors 65, 70 to form a fibrous insulation blanket 10. It is to be appreciated that the insulation blanket 10 has an upper surface and a lower surface. In particular, the insulation blanket 10 has two major surfaces, typically a top and bottom surface, and two minor or side surfaces with fiber blanket 10 oriented so that the major surfaces have a substantially horizontal orientation. The upper and lower oven conveyors 65,

70 may be used to compress the insulation pack 40 to give the insulation blanket 10 a predetermined thickness. It is to be appreciated that although FIG. 1 depicts the conveyors 65, 70 as being in a substantially parallel orientation, they may alternatively be positioned at an angle relative to each other (not illustrated).

[0025] The curing oven 60 may be operated at a temperature from about 100 °C to about 325 °C, or from about 250 °C to about 300 °C. The insulation pack 40 may remain within the oven for a period of time sufficient to drive off excess water and to crosslink (cure) the binder and form the insulation blanket 10.

[0026] A facing material 93 may optionally be placed on the insulation blanket 10 to form a facing layer 95. It should be appreciated that a flexible “blanket” insulation product is depicted in Fig. 1, but the insulation product may also be a rigid panel or board-type insulation product. Non-limiting examples of suitable facing materials 93 include fiberglass mats, Kraft paper, a foil-scrim-Kraft paper laminate, recycled paper, and calendered paper. The facing material 93 may be adhered to the surface of the insulation blanket 10 (board) by a bonding agent (not shown in Fig 1) to form a faced insulation product 97. When sufficiently flexible, the faced fibrous insulation 97 may subsequently be rolled for storage and/or shipment or cut into predetermined lengths by a cutting device (not illustrated). Such faced insulation products may be used, for example, as panels in basement finishing systems, as ductwrap, ductboard, as faced residential insulation, as construction panels for walls and ceilings, and as pipe insulation.

[0027] Fibrous products are generally formed of matted fibers, often bonded together by a cured thermoset or thermoplastic polymeric material. Examples of suitable fibers include mineral fibers such as glass fibers, wool glass fibers, rock, basalt, slag and ceramic fibers. For example, the glass fibers may be produced from a variety of natural minerals or manufactured chemicals such as silica sand, limestone, and soda ash. Other ingredients may include calcined alumina, borax, feldspar, nepheline syenite, magnesite, and kaolin clay. Optionally, other fibers such as natural fibers and/or synthetic fibers such as polyester, polyethylene, polyethylene terephthalate, polypropylene, polyamide, polyvinyl alcohol, aramid, and/or polyaramid fibers may be present in the insulation product in addition to the glass fibers. The term “natural fiber” as used in conjunction with the present invention refers to plant fibers extracted from any part of a plant, including, but not limited to, the stem, seeds, leaves, roots, or phloem. Examples of natural fibers suitable for use as the reinforcing fiber material include cellulose, basalt, cotton, jute, bamboo, ramie, bagasse, hemp, coir, linen, kenaf, sisal, flax, henequen, and combinations thereof. Insulation products may be

formed entirely of one type of fiber, or they may be formed of a combination of types of fibers. For example, the insulation product may be formed of combinations of various types of glass fibers or various combinations of different inorganic fibers and/or natural fibers depending on the desired application for the insulation. While other natural, polymeric and mineral fibers are known, the embodiments described herein are primarily with reference to glass fiber insulation products.

[0028] The term “fibrous products” is general and encompasses a variety of articles of manufacture. This has already been noted and is evident from table B, below. “Fibrous products” may be characterized and categorized by many different properties, one of which is density. Density may range broadly from about 0.2 pounds/cubic foot (“pcf”) to as high as about 10 pcf, depending on the product. Low or light density insulation batts and blankets typically have densities between about 0.2 pcf and about 5 pcf, more commonly from about 0.3 to about 4 pcf, and have applications rates of about 2-13% LOI. Products such as residential insulation batts may fall in this group.

[0029] Fiberglass insulation products can be provided in other forms including board (a heated and compressed batt) and molding media (an alternative form of heated and compressed batt) for use in different applications. Fibrous products also include higher density products having densities from about 10 to about 20 pcf, (and often having binder LOI in excess of 12%) and medium density products more typically having a density from about 1 pcf to about 10 pcf, (and having binder LOI of about 7-16 wt% LOI) such as boards and panels. Medium and higher density insulation products may be used in industrial and/or commercial applications, including but not limited to metal building insulation, pipe or tank insulation, insulative ceiling and wall panels, duct boards and HVAC insulation, appliance and automotive insulation, etc.

[0030] Another property useful for categorization is the rigidity of the product. Residential insulation batts are typically quite flexible and they can be compressed into rolls or batts while recovering their “loft” upon decompression. In contrast, other fibrous products, such as ceiling tiles, wall panels, foundation boards and certain pipe insulation to mention a few, are quite rigid and inflexible by design. These products will flex very little and are unlikely to be adapted or conformed to a particular space.

[0031] Formed or shaped products may include a further step, optionally during cure, that compresses, molds or shapes the product to its specific final shape. Rigid boards are a type of shaped product, the shape being planar. Other shaped products may be formed by dies or molds or other forming apparatus. Rigidity may be imparted by the use of higher density of

fibers and/or by higher levels of binder application. As an alternative to rotary fiberizing, some fibrous insulation products, particularly higher density, non-woven insulation products, may be manufactured by an air-laid or wet-laid process using premade fibers of glass, other minerals or polymers that are scattered into a random orientation and contacted with binder to form the product.

[0032] Some exemplary fibrous products that can be manufactured according to the invention include those illustrated in Table A below.

[0033] Table A: Bio-based binder formulations for representative products*

	Flexible Duct Media	Metal Building Insulation	Warm & Dry boards	Ceiling Tile boards		
				A	B	C
Maltodextrin	65-70	65-70	65-70	65-70	45-60	10-50
Citric Acid	25-30	25-30	25-30	25-30	30-35	20-40
Polyacrylic Acid						10-60
Sodium hypophosphite	2-5	2-5	2-5	2-5	2-5	2-10
Glycerol					10-15	5-15
Surfactant (e.g. SURFYNOL 465)	0 - 0.5	0 - 0.5	0.1 – 0.5	0.1 – 0.5	0.1 – 0.5	0.1 – 0.5
Reactive polysiloxane	0.01 -5.0	0.01 -5.0	0.4-3.0	0.01 -3.0	0.01 -3.0	0.01 -3.0

*In Table A above, each ingredient of the binder composition is given as a range of typical values of percentage of dry weight of the binder composition.

[0034] A further listing of insulation fibrous products that can be manufactured using a bio-based binder composition according to the invention is set forth in Table B, below.

Insulation products such as ceiling tiles, wall panels and other construction panels may have having finished facings layers 93. These products generally have finished surfaces designed to be the outermost layer of ceilings or walls in buildings such as homes, offices, etc.

TABLE B: Selected Commercial and Industrial Fibrous Products which may use a Bio-Based Binder

	Rigid Boards	Flexible, Light Density Insulation	Rigid Pipe Insulation and pipe rolls	Textile E-glass Nonwoven
Density	Wide range of densities- from 1.5 to 10 pcf	Light density – Ranging from 0.3 to 4.0 pcf	Ranging from 3-6 pcf	Ranging from 0.8 to 4 pcf
Binder content	about 2 to about 20% LOI	about 2 to about 13% LOI	about 3 to about 15% LOI	about 5 to about 20% LOI
Manufacturing method	Rotary fiber forming process	Rotary fiber forming process	Rotary fiber forming process plus on or offline molding/ pipe formation process	Air-laid nonwoven process
Exemplary Owens Corning Products	• QUIET R Duct Board • QUIET R Duct Liner Board • 700 Series Insulation • Insul-Quick Insulation • SCR Insulation Board • Curtainwall • QuietZone Shaftwall • Warm -N -Dri • Energy Board • TremDrain • Exterior Foundation • Barrier Board • Ceiling Board Blanks	• Certified R Metal Building Insulation • ELAMINATOR® Pre-Engineered Metal Roof Insulation • MBI Plus • Metal Bldg Utility Blanket • Unfaced Metal Building Insulation for Canada • Flexible Duct Media Insulation • QUIET R Rotary Duct Liner • SOFTR Duct Wrap FRK • TIW Types I and II • FLEX-Wrap for pipes and tanks • H2V Series • RA Series • Select Sound • Thermorange • FlameSpread 25 • Sonobatts • Thermal Batts	• EVOLUTION Paper-Free ASJ • VAPORWICK Insulation • FIBERGLAS™ Pipe and Tank Insulation rolls	• QUIET R Textile Duct Liner • DURAFLEX Transportation

[0034] “Product properties” or “mechanical properties” refers to a battery of testable physical properties that insulation products possess. These may include at least the following common properties:

- “Recovery” – which is the ability of the batt or blanket to resume its original or designed thickness following release from compression during packaging or storage. It may be tested by measuring the post-compression height of a product of known or intended nominal thickness, or by other suitable means.
- “Restoring Force” – which is like Recovery in measuring the batt’s ability to resume its original thickness. However, for Restoring Force, the height of expansion is restricted and the force exerted by expansion is measured by a scale or other force measuring device or gauge.
- “Stiffness” or “sag” – which refers to the ability of a batt or blanket to remain rigid and hold its linear shape. It is measured by draping a fixed length section over a fulcrum and measuring the angular extent of bending deflection, or sag. Lower values indicate a stiffer and more desirable product property. Other means may be used.
- “Tensile Strength” – which refers to the force that is required to tear the fibrous product in two. It is typically measured in both the machine direction (MD or X-axis) and in the cross machine direction (“CD” or “XMD” or Y-axis); and sometimes in a depth or Z-axis direction as well.
- “Compressive Strength” – which refers to the force that is required compress the fibrous insulation product. This may be measured as the force required to compress the batt (or package) a predetermined distance, or as the distance compressed by a predetermined force. It may be measured in any of three directions as with tensile strength, but CD is most typical.
- “Lateral weight distribution” (LWD or “cross weight”) – which is the relative uniformity or homogeneity of the product throughout its width. It may also be thought of as the uniformity of density of the product, and may be measured by sectioning the product longitudinally into bands of equal width (and size) and weighing the band, by a nuclear density gauge, or by other suitable means.
- “Vertical weight distribution” (VWD) – which is the relative uniformity or homogeneity of the product throughout its thickness. It may also be thought of as the uniformity of density of the product, and may be measured by sectioning the product

horizontally into layers of equal thickness (and size) and weighing the layers, by a nuclear density gauge, or by other suitable means.

[0035] Of course, other product properties may also be used in the evaluation of final product, but the above product properties are ones found important to consumers of insulation products. Mechanical product properties may be tested relatively soon after manufacture – a time referred to herein as “initial” or “ambient” even if it is days or weeks due to shipping lag. But over time, the mechanical properties may degrade so that a more relevant test is one that measures “aged” mechanical properties. Aging may be natural, real-time aging over the course of several months or years. More typically “aging” is simulated in proxy, accelerated aging conditions, as in the case of hot and humid test conditions. While either type of aging produced “aged” properties that can be measured, the accelerated versions are reasonable proxies that can be tested in a matter of days rather than months.

[0036] It should be appreciated that, to some extent, the absolute measures of these mechanical product properties may be dependent on how much binder is applied to the fibers. Denser and more rigid products are typically manufactured, in part, by using higher levels of binder. The measure of how much binder is applied to glass fiber products is known as LOI, or loss on ignition, measured by the weight difference after burning off the organic binder components.

[0037] In some cases it is desirable to compute a percent improvement in a mechanical property. This is calculated as the difference in the measured property divided by the property in the control product. Depending on the specific property and the level of loading of the surfactant in accordance with the invention, one finds improvement of mechanical properties of at least 2%, at least 5%, at least 10%, at least 15%, at least 20% or at least 25%.

Binder Compositions

[0038] Binder compositions are well known in the industry. Binders are typically applied to the fibers as an aqueous solution or dispersion shortly after the fibers are formed and then cured at elevated temperatures. The curing conditions are selected both to evaporate any remaining solvent and cure the binder to a thermoset state. The fibers in the resulting product tend to be partially coated with a thin layer of the thermoset resin and exhibit accumulations of the binder composition at points where fibers touch or are positioned closely adjacent to each other. In one embodiment, phenol-formaldehyde binders are used with polysiloxane additives to provide increased water and stain resistance to the insulation product. Phenol-

formaldehyde binders are generally characterized by relatively low viscosity when uncured, and the formation of a rigid thermoset polymeric matrix with the fibers when cured. A low-viscosity uncured binder simplifies binder application and allows the binder-coated batt to expand more easily when the forming chamber compression is removed. Similarly, the rigid matrix formed by curing the binder allows a finished fiber product to be compressed for packaging and shipping and then recover to substantially its full original dimension when unpacked for installation. As used herein, "dispersion" includes all forms of solids dispersed in a liquid medium, regardless of the size of the particle or properties of the dispersion, including true "solutions" in which the solids are soluble and dissolved in the liquid medium.

[0039] In other embodiments, formaldehyde-free binders may also be used in combination with additives that increase the insulation products resistance to water. Nonphenol/ formaldehyde binders exhibit low uncured viscosity and structural rigidity when cured. One such binder composition is disclosed in U.S. Pat. No. 5,318,990, which is herein incorporated, in its entirety, by reference, and utilizes a polycarboxy polymer, a monomeric trihydric alcohol and a catalyst comprising an alkali metal salt of a phosphorous containing organic acid. Other binder compositions have also been developed to provide reduced emissions during the coating and curing processes utilizing compounds such as polyacrylic acid as disclosed in U.S. Pat. Nos. 5,670,585 and 5,538,761, which are herein incorporated, in their entirety, by reference.

[0040] Although the invention may be employed with traditional phenol-formaldehyde (PF) or phenol-urea-formaldehyde (PUF) binders, in other embodiments, the invention is employed with formaldehyde-free binders, such as polyacrylic acid binders utilizing as described in U.S. Patents 6,884,849 and 6,699,945 to Chen, et al. Another polyacrylic binder composition is disclosed in U.S. Pat. No. 5,661,213, which teaches an aqueous composition comprising a polyacid, a polyol and a phosphorous-containing accelerator, wherein the ratio of the number of equivalents of the polyacid to the number of equivalents of the polyol is from about 100: 1 to about 1:3 and is hereby fully incorporated by reference. As disclosed in U.S. Pat. No. 6,399,694, which hereby also fully incorporated by reference, another alternative to the PUF binders utilizes polyacrylic acid and either glycerol (PAG) or triethanolamine (PAT) as a binder. PAG/PAT binders are relatively odorless, more uniformly coat each fiber and have a generally white or light color.

[0041] Also useful are binders made from natural starches (or dextrans, maltodextrins or other polysaccharides of varying length) and polyfunctional carboxylic acids like citric acid (MD/CA), such as those disclosed in US2011/0086567 and WO 2011/044490, published

April 14, 2011, all incorporated by reference. These polyhydroxyl-carboxylic acid-based binder systems, however, are also described herein.

Polyhydroxyl compounds

[0042] By definition, the polyhydroxy compound or polyol is polyvalent, having two or more hydroxyl groups that can be available for reaction. While a polyol has a minimum of two hydroxyl groups, there is no theoretical maximum number of hydroxyl groups. Diols, triols, tetraols, penta-ols, hexa-ols and higher polyols are all encompassed, particularly in polymeric compounds. The polyol may be monomeric or polymeric; and may be natural or synthetic. In some embodiments, the polyol may be smaller monomeric compounds like glycerol, ethylene glycol, propanediols, propanetriols, trimethylol propane, erythritol or other butane-based polyols, pentaerythritol, triethanolamine (TEOA), or 1,2,6- hexane-triol; or any monosaccharide having at least 4 carbons, including pentoses and hexoses.

[0043] In other embodiments, the polyol may be a synthetic or naturally occurring polymer, such as polyvinyl alcohol, polyglycerol, poly(ether) polyols, poly(ester) polyols, polyethylene glycol, polyol- and hydroxy-functional acrylic resins such as JONCRYL® (BASF Resins), MACRYNAL® (Cytec Industries) PARALOID® (Dow Coating Materials), G-CURE®, TSAX® and SETALUX® (Nuplex Resins, LLC) in solution or emulsion form; or di-, tri- and higher polysaccharides.

[0044] Due to the wide variability in molecular weights of the polyol component and (as discussed below) the crosslinking agent, the weight ratios of the various components of the binder composition can vary tremendously. Thus, polyol (polyhydroxyl) component may be present in the binder composition in an amount from about 1% to about 99% by weight of the total solids in the binder composition, more likely from about 20% to about 99% by weight of the total solids in the binder composition. As is common in the industry and as used herein, % by weight indicates % by weight of the total solids (i.e. dry weight, without water) in the binder composition. For purposes of this application, this is synonymous with “% binder solids” and “% total solids” where discussing binder dispersions.

[0045] In some exemplary embodiments, the polyol component is a carbohydrate, such as a starch or maltodextrin, and the binder further includes a crosslinking agent. In some exemplary embodiments, the carbohydrate-based binder composition also includes a coupling agent, a process aid agent, an extender, a pH adjuster, a catalyst, a crosslinking density enhancer, a deodorant, an antioxidant, a dust suppressing agent, a biocide, a moisture resistant agent, a surfactant, or combinations thereof. The binder may be used in the

formation of many insulation materials, including but not limited to batts, rolls, construction panels, etc. In addition, the binder is free of added formaldehyde. Further, the binder composition has a reduction in particulate emission compared to conventional phenol/urea/formaldehyde binder compositions. The inventive binder may also be useful in forming particleboard, plywood, and/or hardboards.

[0046] In one or more exemplary embodiments, the binder includes at least one polyol that is natural in origin and derived from renewable resources. For instance, the polyol may be a carbohydrate derived from plant sources such as legumes, maize, corn, waxy corn, sugar cane, milo, white milo, potatoes, sweet potatoes, tapioca, rice, waxy rice, peas, sago, wheat, oat, barley, rye, amaranth, and/or cassava, as well as other plants that have a high starch content. As is well known in the arts, starches can be degraded into a wide variety of polysaccharides of various length, molecular weight and other properties, specifically including but not limited to dextrans, maltodextrins, and syrups of varying conversion from low to very high. The carbohydrate polymer may also be derived from crude starch-containing products derived from plants that contain residues of proteins, polypeptides, lipids, and low molecular weight carbohydrates.

[0047] As noted, the carbohydrate may be selected from monosaccharides, including but not limited to erythrose, erythulose, threose, ribose, ribulose, arabinose, xylose, xylulose, glucose, dextrose (or D-glucose), mannose, galactose, fructose, and sorbose; disaccharides, including but not limited to maltose, sucrose, lactose, cellobiose and trehalose; oligosaccharides (*e.g.*, glucose syrup and fructose syrup); and polysaccharides (*e.g.*, pectin, dextrin, maltodextrin, starch, modified starch, and starch derivatives), provided they can be prepared as water dispersions, which includes emulsions, suspensions, colloids and true solutions. All isomeric and stereochemical forms of these saccharides are encompassed in the invention. Furthermore, derivatives of saccharides may also be suitable, provided they retain their polyvalent polyol nature after derivatization. Thus, the polyol may include O-glycosides, N-glycosides, S-glycosides, C-glycosides, O-alkyl (*e.g.* methyl, ethyl), O-acylated sugars, amino sugars, sugar alcohols (like sorbitol, xylitol, erythritol, etc.) and the like.

[0048] The carbohydrate polymer may have a number average molecular weight from about 1,000 to about 8,000. Additionally, the carbohydrate polymer may have a dextrose equivalent (DE) number from 2 to 20, from 5 to 15, or from 7 to 12. The carbohydrate dispersions beneficially have a low viscosity and cure at moderate temperatures (*e.g.*, 80-250 °C) alone or with additives. The low viscosity enables the carbohydrate to be utilized in a

binder composition. The use of a carbohydrate in the inventive binder composition is advantageous in that carbohydrates are readily available or easily obtainable and are low in cost.

[0049] In at least one exemplary embodiment, the carbohydrate is a water-soluble polysaccharide such as dextrin or maltodextrin. The carbohydrate polymer may be present in the binder composition in an amount from about 20% to about 90% by weight of the total solids in the binder composition, from about 45% to about 85% by weight of the total solids in the binder composition, from about 50% to about 80%, or from about 55% to about 75%.

[0050] It will be understood that mixtures or blends of two or more polyhydroxyl compounds of the same or different type may be used in a binder composition. For example, but not as a limitation, blends of any of the following may be envisioned:

- Two different monosaccharides, such as glucose with fructose or sorbose;
- Two different polysaccharides, such as dextrin and a maltodextrin or syrup;
- A polysaccharide and a mono- or oligosaccharide;
- A polysaccharide and a synthetic polyol such as glycerol; and
- A mono- or oligosaccharide with a synthetic polyol such as glycerol.

Polycarboxylic acid crosslinking agents

[0051] In addition, the binder composition contains a polycarboxylic acid crosslinking agent suitable for crosslinking the polyhydroxyl compound. In exemplary embodiments, the crosslinking agent has a number average molecular weight greater than 90, from about 90 to about 10,000, or from about 190 to about 4,000. In some exemplary embodiments, the crosslinking agent has a number average molecular weight less than about 1000. Non-limiting examples of suitable crosslinking agents include di-, tri- and polycarboxylic acids (and salts thereof), anhydrides, monomeric and polymeric polycarboxylic acid with anhydride (*i.e.*, mixed anhydrides), malonic acid, succinic acid, glutaric acid, maleic acid, citric acid (including salts thereof, such as ammonium citrate), 1,2,3,4-butane tetracarboxylic acid, adipic acid, polyacrylic acid, and polyacrylic acid based resins such as QXRP 1734, 1629 and Acumer 9932, all commercially available from The Dow Chemical Company. In exemplary embodiments, the crosslinking agent may be any monomeric or polymeric polycarboxylic acid, citric acid, and their corresponding salts. For each type of acid, it should be understood that acid salts may also be used in place of the acids. It should also be understood that mixtures or blends of two or more different polycarboxylic acids may be used.

[0052] The nomenclature of the polycarboxylic acid as the crosslinking agent is somewhat arbitrary. The polyhydroxyl compound and polycarboxylic acid react and it is a matter of convenience to think of the typically smaller polycarboxylic acid as crosslinking the typically larger polyhydroxyl polymer. However, it is equally plausible to consider a larger polymeric polycarboxylic acid that is crosslinked by a smaller polyhydroxyl molecule (e.g. glycerol or TEOA).

[0053] The crosslinking agent may be present in the binder composition in an amount up to about 50% by weight of the binder composition. In exemplary embodiments, the crosslinking agent may be present in the binder composition in an amount from about 20% to about 40% by weight of the total solids in the binder composition or from about 25% to about 35% by weight.

[0054] It should be understood that application of surfactant and/or optional silane coupling agent, as described in sections below, may conveniently be done through incorporation of those agents into a binder dispersion. Consequently, the concentration of these agents is often expressed on the basis of a binder composition weight. However, this is not the only means for applying surfactant or silane to the fibrous materials. Other known spray nozzles, dispensers, or roll coating operations are also suitable mechanisms for applying these agents to the mineral fibers.

Surfactants

[0055] Surfactants have been used in fibrous products – generally as a component of a binder composition – as a wetting agent to promote the distribution of binder within the spray systems and throughout the fibrous product (see e.g. WO 2011/044490). Surfactants are available in a wide variety of configurations for different purposes. In general, surfactants have a polar region, or head, and a non-polar region, or tail. This provides them with a portion that likes water or other polar solvents; and a portion that likes oils or non-polar solvents. Due to their dual functionality (both polar and non-polar) surfactants generally reside at the interfaces between dissimilar media, such as at water-oil interfaces or water-air interfaces. In this way, they can reduce the surface tension at these interfaces. After a certain surfactant concentration is reached however, the interfaces are saturated with surfactant molecules and additional surfactant will form micelles in the medium rather than crowding in at the interface. This concentration is known as the critical micelle concentration (“CMC”) and is typically published for each surfactant. Above this CMC, surface tension is not significantly reduced.

[0056] Surfactants may be classified into groups, in part, based on the degree or magnitude of this affinity for polar solvents (hydrophilicity) or for non-polar solvents (lipophilicity) to produce a HLB number (known as the hydrophilic lipophilic balance) of the surfactant. The polar head may be neutral (non-ionic) or charged (e.g. anionic(-), cationic(+) or zwitterionic (both)), which provides a way to classify surfactants based on electronic charge. Electronic charge may be dependent on pH however. Finally, the chemical structure of the surfactants provides yet another way to classify surfactants, such as the “alkyl sulfates” or “quaternary ammonium salts” or ethoxylated or polyethoxylated alcohols or ethers.

[0057] Neutral or non-ionic surfactants have been found suitable for the present invention, including for example the alkyl glucosides, alkyl thioglucosides, ethoxylated alcohols, ethoxylated ethers, the polyoxyethylenes (e.g. TRITON X™, PLURONICS™, BRIJ™ and TWEEN™ series), ethylene oxide, and 1,4 dioxane. Non-limiting examples of suitable surfactants include the Surfynol® series 420, 440, 465, and 485, which are ethoxylated 2,4,7,9-tetramethyl-5-decyn-4,7-diol surfactants (commercially available from Air Products and Chemicals, Inc. (Allentown, PA)); Triton™ X-100 and Triton™ X-405, which are polyethoxylated p-(1,1,3,3-tetramethylbutyl) phenyl ethers (sold commercially by The Dow Chemical Co. (Midland, MI)); Polysorbate 20 (ethoxylated sorbitan monolaurate) and Polysorbate 80 (ethoxylated sorbitan monooleate) sold commercially by Sigma-Aldrich (St. Louis, MO); and sulfates (e.g., alkyl sulfates, ammonium lauryl sulfate, sodium lauryl sulfate (SDS), alkyl ether sulfates, sodium laureth sulfate, and sodium myreth sulfate

[0058] The surfactant may be present in the binder composition in a concentration from about 0.01 % to about 10% by weight of the total solids in the binder composition, and all subranges within this range. For example, in various embodiments, the surfactant is from about 0.01 % to about 5.0% by weight, or from about 0.05 % to about 1 % by weight, or from about 0.05 % to about 0.5% by weight, or from about 0.1 % to about 0.5% by weight all based on the total solids in the binder. The surfactant concentration in the fibrous product may be calculated as the percent in the binder times the application rate of binder or LOI (loss on ignition- which is an assay for binder concentration on glass fibers). Target LOI varies greatly depending on the specific product as noted in table B above; for example from about 2% to 20% or more, which gives a broad range of very small absolute concentrations of surfactant in product. For example, a 0.1% binder dispersion applied with a target LOI of 6% results in a calculated final product concentration of just 0.006%. While possible to calculate these small numbers, applicants define instead a “normalized” surfactant concentration in product which is the absolute concentration divided by the LOI. As with

some measures of mechanical properties, this normalization vs LOI is a fairer way to compare the surfactant content of products with differing binder LOI levels. Mathematically, the “normalized” surfactant concentration of a fibrous product is the same as the surfactant concentration of the binder that is used to manufacture it; multiplying by the LOI and then dividing by it cancels this term. Those skilled in the art will recognize that the CMC of any specific surfactant can further inform the decision regarding concentration in the upper ranges of concentrations given above.

Silane coupling agent

[0059] Silane coupling agents or organosilanes are well known in the glass fiber forming industry. They have been used in sizings for protection of long filaments, and as additives to binder compositions. It is thought that the silane portion bonds to silica of the glass, and the organic portion helps to bind or hold resins, binders or other organic materials to the glass. Organosilanes have thus thought to provide a protective function for glass fibers, although they are optional in the present invention. In some embodiments of the invention, a silane coupling agent may optionally be used in combination with a surfactant to improve the aged mechanical properties of a fibrous insulation product

[0060] Non-limiting examples of silane coupling agents that may optionally be used in the binder composition may be characterized by the functional groups alkyl, aryl, amino, epoxy, vinyl, methacryloxy, ureido, isocyanato, and mercapto. In exemplary embodiments, the silane coupling agent(s) include silanes containing one or more nitrogen atoms that have one or more functional groups such as amine (primary, secondary, tertiary, and quaternary), amino, imino, amido, imido, ureido, or isocyanato. Specific, non-limiting examples of suitable silane coupling agents include, but are not limited to, aminosilanes (e.g., 3-aminopropyl-triethoxysilane and 3-aminopropyl-trihydroxysilane), epoxy trialkoxysilanes (e.g., 3-glycidoxypropyltrimethoxysilane and 3-glycidoxypropyltriethoxysilane), methacryl trialkoxysilanes (e.g., 3-methacryloxypropyltrimethoxysilane and 3-methacryloxypropyltriethoxysilane), hydrocarbon trialkoxysilanes, amino trihydroxysilanes, epoxy trihydroxysilanes, methacryl trihydroxy silanes, and/or hydrocarbon trihydroxysilanes. In one or more exemplary embodiments, the silane is an aminosilane, such as γ -aminopropyltriethoxysilane.

[0061] Further exemplary coupling agents (including silane coupling agents) suitable for use in the binder composition are set forth below:

- Acryl: 3-acryloxypropyltrimethoxysilane; 3-acryloxypropyltriethoxysilane; 3-acryloxypropylmethyldimethoxysilane; 3-acryloxypropylmethyldiethoxysilane; 3-methacryloxypropyltrimethoxysilane; 3-methacryloxypropyltriethoxysilane
- Amino: aminopropylmethyldimethoxysilane; aminopropyltriethoxysilane; aminopropyltrimethoxysilane/EtOH; aminopropyltrimethoxysilane; N-(2-aminoethyl)-3-aminopropyltrimethoxysilane; N-(2-aminoethyl)-3-aminopropylmethyldimethoxysilane; (2-aminoethyl)-(2-aminoethyl) 3-aminopropyltrimethoxysilane; N-phenylaminopropyltrimethoxysilane
- Epoxy: 3-Glycidoxypropylmethyldiethoxysilane; 3-glycidoxypropylmethyldimethoxysilane; 3-glycidoxypropyltriethoxysilane; 2-(3,4-epoxycyclohexyl)ethylmethyldimethoxysilane; 2-(3,4-epoxycyclohexyl)ethylmethyldiethoxysilane; 2-(3,4-epoxycyclohexyl)ethyltrimethoxysilane; 2-(3,4-Epoxy-cyclohexyl)ethyltriethoxysilane
- Mercapto: 3-mercaptopropyltrimethoxysilane; 3-Mercaptopropyltriethoxysilane; 3-mercaptopropylmethyldimethoxysilane; 3-Mercaptopropylmethyldiethoxysilane
- Sulfide: bis[3-(triethoxysilyl)propyl]-tetrasulfide; bis[3-(triethoxysilyl)propyl]-disulfide
- Vinyl: vinyltrimethoxysilane; vinyltriethoxysilane; vinyl tris(2-methoxyethoxy)silane; vinyltrichlorosilane; trimethylvinylsilane
- Alkyl: methyltrimethoxysilane; methyltriethoxysilane; dimethyldimethoxysilane; dimethyldiethoxysilane; tetramethoxysilane; tetraethoxysilane; ethyltriethoxysilane; n-propyltrimethoxysilane; n-propyltriethoxysilane; isobutyltrimethoxysilane; hexyltrimethoxysilane; hexyltriethoxysilane; octyltrimethoxysilane; decyltrimethoxysilane; decyltriethoxysilane; octyltriethoxysilane; tert-butyl dimethylchlorosilane; cyclohexylmethyldimethoxysilane; dicyclohexyldimethoxysilane; cyclohexylethyldimethoxysilane; t-butylmethyldimethoxysilane
- Chloroalkyl: 3-chloropropyltriethoxysilane; 3-chloropropyltrimethoxysilane; 3-chloropropylmethyldimethoxysilane
- Perfluoro: decafluoro-1,1,2,2-tetrahydrodecyl)trimethoxysilane; ((heptadecafluoro-1,1,2,2-tetrahydrodecyl)trimethoxysilane
- Phenyl: phenyltrimethoxysilane; phenyltriethoxysilane; diphenyldiethoxysilane; diphenyldimethoxysilane; diphenyldichlorosilane
- Hydrolyzates of the silanes listed above
- Zirconates: zirconium acetylacetonate; zirconium methacrylate

- Titanates: tetra-methyl titanate; tetra-ethyl titanate; tetra-n-propyl titanate; tetra-isopropyl titanate; tetra-isobutyl titanate; tetra-sec-butyl titanate; tetra-tert-butyl titanate; mono n-butyl, trimethyl titanate; mono ethyl tricyclohexyl titanate; tetra-n-amyl titanate; tetra-n-hexyl titanate; tetra-cyclopentyl titanate; tetra-cyclohexyl titanate; tetra-n-decyl titanate; tetra n-dodecyl titanate; tetra (2-ethyl hexyl) titanate; tetra octylene glycol titanate ester; tetrapropylene glycol titanate ester; tetra benzyl titanate; tetra-p-chloro benzyl titanate; tetra 2-chloroethyl titanate; tetra 2-bromoethyl titanate; tetra 2-methoxyethyl titanate; tetra 2-ethoxyethyl titanate.

[0062] The coupling agent(s), when present, may be present in the binder composition in an amount from about 0.01% to about 5.0% by weight of the total solids in the binder composition, from about 0.01% to about 2.5% by weight, or from about 0.1% to about 0.5% by weight. Absolute silane coupling agent concentrations in product may be calculated as described above for surfactants, however, the “normalized” concept is useful here again.

Other optional ingredients of the Binder Compositions

[0063] Typically, the binder composition may include a cure accelerator or catalyst to assist in the crosslinking. A cure accelerator may be consumed in the reaction whereas a pure catalyst is not. As used herein the term “catalyst” encompasses cure accelerators as well as pure catalysts. The catalyst may include inorganic salts, Lewis acids (*i.e.*, aluminum chloride or boron trifluoride), Bronsted acids (*i.e.*, sulfuric acid, *p*-toluenesulfonic acid and boric acid) organometallic complexes (*i.e.*, lithium carboxylates, sodium carboxylates), and/or Bronsted or Lewis bases (*i.e.*, polyethyleneimine, diethylamine, or triethylamine). Additionally, the catalyst may include an alkali metal salt of a phosphorous-containing organic acid; in particular, alkali metal salts of phosphorus acid, hypophosphorus acid, or polyphosphoric acids. Examples of such phosphorus catalysts include, but are not limited to, sodium hypophosphite, sodium phosphate, potassium phosphate, disodium pyrophosphate, tetrasodium pyrophosphate, sodium tripolyphosphate, sodium hexamethaphosphate, potassium phosphate, potassium tripolyphosphate, sodium trimetaphosphate, sodium tetramethaphosphate, and mixtures thereof. In addition, the catalyst may be a fluoroborate compound such as fluoroboric acid, sodium tetrafluoroborate, potassium tetrafluoroborate, calcium tetrafluoroborate, magnesium tetrafluoroborate, zinc tetrafluoroborate, ammonium tetrafluoroborate, and mixtures thereof. Further, the catalyst may be a mixture of phosphorus and fluoroborate compounds. Other sodium salts such as, sodium sulfate, sodium nitrate, sodium carbonate may also or alternatively be used as the catalyst, as well as some lithium

and zirconium complexes. Carbodiimide based coupling agents like and not limited to 1-ethyl-3-(3-dimethylaminopropyl) carbodiimide (EDCI) or N,N'-Dicyclohexylcarbodiimide (DCC) could be used as well. The catalyst may be present in the binder composition in an amount from about 0% to about 10% by weight of the total solids in the binder composition, or from about 1.0% to about 5.0% by weight, or from about 3.0% to about 5.0% by weight.

[0064] Binder compositions may contain reactive polysiloxanes as moisture resistance agents, lubricants or for other purposes. Reactive polysiloxanes (also known as reactive silicones) are silicon polyethers having at least one of the typical alkyl substituents of inert silanes replaced by a reactive functional group. For purposes of this invention in the context of a reactive polysiloxane, a "reactive functional group" includes hydride (-H), hydroxyl (-OH), amino (-NH₂), carboxyl (-COOH). Polysiloxanes having a reactive hydrogen are among the most common and are referred to as polyalkylhydrogensiloxanes. Polysiloxanes having a reactive amino group are polyalkylaminosiloxanes; polysiloxanes having a reactive hydroxyl group are polyalkylhydroxylsiloxanes; and polysiloxanes having a reactive carboxyl group are polyalkylcarboxylsiloxanes; regardless whether the reactive functional group is attached directly to the silicon atom or to a lower alkyl or aryl attached to the silicon atom.

[0065] In addition, the binder composition may include a processing aid to facilitate the processing of the fibers formation and orientation. Examples of processing aids include viscosity modifiers, defoaming agents and lubricants. Additionally, binder compositions may optionally include; a biocide; a crosslinking density enhancer to improve the degree of crosslinking; organic and/or inorganic acids and bases in an amount sufficient to adjust and/or buffer the pH to a desired level; a moisture resistant agent; a dust suppressing agent to reduce or eliminate the presence of inorganic and/or organic particles; fillers or extenders to improve the binder's appearance and/or to lower the overall manufacturing cost; and conventional additives such as, but not limited to dyes, pigments, colorants, UV stabilizers, thermal stabilizers, lubricants, anti-foaming agents, anti-oxidants, emulsifiers, preservatives (*e.g.*, sodium benzoate), corrosion inhibitors, and mixtures thereof.

EXAMPLES

[0066] The following examples serve to further illustrate the invention.

Example 1: Preparation of R-19 fibrous batts

[0067] Fibrous insulation batts designated with R-value 19 are prepared by rotary fiberization and coated with a maltodextrin – citric acid binder (70:30 ratio) containing oil,

0.18% gamma-aminopropyltrihydroxy silane and varying amounts of a surfactant (Surfynol® 465), expressed herein as a percent of binder solids. Binder was applied at a target level of 6.3 % LOI, making the calculated amount of surfactant in the fibrous product as shown in Table 1 below. The normalized concentration of surfactant in the fibrous product is the same as the concentration of surfactant in the binder composition.

[0068] Table 1: Surfactant concentrations

Surfactant as % of binder solids in binder composition	Surfactant as % of fibrous product (assuming 6.3% LOI)
0.09	0.0056
0.27	0.0168
0.44	0.0279

Example 2: Testing R-19 batts for Sag/Stiffness

[0069] R-19 fibrous batts prepared as in Example 1 are exposed to an accelerated hot and humid treatment conditions designed to be a proxy for real-time aging in warm and humid climates. The proxy test exposes the batts to 90°F and 90% relative humidity (RH) for 3 days. Sag or stiffness is a mechanical product property that indicates the strength of the fibrous batt as a fixed length is draped over a fulcrum. The degree to which its ends deviate from a straight line constitutes the sag. Sag was tested both before (Ambient) and after (H/H) hot and humid test conditions. The results of N=120 measurements are depicted in Fig. 2 which includes 95% confidence interval (CI) bars about the means, as well as a linear trend line; and in Table 2 below

[0070] Table 2: Sag/Stiffness (degrees) and percent change vs no surfactant

%Surfynol	Avg Sag (deg) under		% improvement under	
	Ambient	Hot/Humid	Ambient	Hot/Humid
0.0	25.38	38.07	--	--
0.09	24.84	31.60	2.1%	17.0%
0.27	24.79	27.77	2.3%	27.1%
0.44	24.94	27.69	1.7%	27.3%

[0071] The results show that with no surfactant, the aged H/H product performed considerably worse than the ambient product, which is typical and not unexpected. But with increasing amounts of surfactant, this disparity grew smaller and smaller. Curiously, increasing amounts of surfactant did not significantly improve the performance of the ambient product. If surfactant were merely functioning as a wetting agent to distribute the binder more uniformly throughout the fibers of the pack, one would expect stronger batts

right from cure (i.e. the ambient curve would be expected to slope downwardly more like the HH curve). Therefore this supposed mechanism does not explain the unexpected HH sag result

Example 3: Testing R-19 batts for Recovery

[0072] R-19 fibrous batts prepared as in Example 1 are exposed to an accelerated hot and humid treatment conditions as described in Example 2. Recovery is a mechanical product property that indicates the ability of the fibrous batt to regain its original or nominal thickness after compression simulating packaging. In this case, nominal thickness was 6.25 inches, for standard R-19 batts. Recovery was tested according to ASTM Std C167-09, both before (ambient) and after (H/H) hot and humid test conditions. The results of N=150 measurements are depicted in Fig. 3, which includes 95% confidence interval (CI) bars about the means, as well as a linear trend line; and in Table 3, below:

[0073] Table 3: Recovery (inches) and percent change vs no surfactant

%Surfynol	Avg Recovery (inches) under		% improvement under	
	Ambient	Hot/Humid	Ambient	Hot/Humid
0.0	6.20	5.65	--	--
0.09	6.08	5.83	-1.9%	3.1%
0.27	6.10	5.80	-1.6%	2.6%
0.44	6.12	5.87	-1.2%	3.9%

[0074] The results show that with no surfactant, the aged H/H product performed considerably worse than the ambient product. But with increasing amounts of surfactant, this disparity grew smaller. Curiously, increasing amounts of surfactant did not significantly improve the performance of the ambient product. If surfactant were merely functioning as a wetting agent to distribute the binder more uniformly throughout the fibers of the pack, one would expect stronger batts right from cure (i.e. the ambient curve would be expected to slope upwardly more like the H/H curve). Therefore this supposed mechanism does not explain the unexpected H/H recovery result.

Example 4: Testing R-30 batts for Sag

[0075] Fibrous insulation batts designated with R-value 30 are prepared by rotary fiberization and coated with the binder of Example 1 having 0.45% of a surfactant (Surfynol® 465). Binder was applied at a target level of 6.5 % LOI, making the calculated amount of surfactant in the fibrous product equal to 0.0289%.

[0076] R-30 batts having a nominal thickness of 9 inches are exposed to an accelerated hot and humid treatment conditions and tested for Sag as described in Example 2 both before

(ambient) and after (H/H) hot and humid test conditions. The results of N=8 measurements are shown in Table 4, below:

[0077] Table 4: R-30 Sag (degrees) and % improvement

	Average Sag (deg)				% improvement	
	Ambient	(CI)	Hot/Humid	(CI)	Ambient	Hot/Humid
control	18.9	+/-1.54	29.0	+/-2.28	--	--
0.45% Surfactant	18.7	+/-1.66	25.3	+/-1.32	1.3%	12.9%

[0078] The data show that 0.45% normalized surfactant in R-30 batts improved the mechanical property of stiffness or sag by more than 10% (nearly 13%) after the aged hot/humid condition treatment. The surfactant modestly improved the sag properties in the ambient condition as well.

Example 5: Testing R-30 batts for Recovery

[0079] R-30 fibrous batts having a nominal thickness of 9 inches are prepared and exposed to an accelerated hot and humid treatment conditions as in Example 4. They are tested for Recovery as described in Example 3 both before (ambient) and after (H/H) hot and humid test conditions. The results of N=200 measurements are shown in Table 4, below:

[0080] Table 5: R-30 Recovery (inches) and % improvement

	Average Recovery (in)				% improvement	
	Ambient	(CI)	Hot/Humid	(CI)	Ambient	Hot/Humid
control	8.9	+/-0.065	8.4	+/-0.061	--	--
0.45% surfactant	8.9	+/-0.061	8.6	+/-0.053	0.9%	1.7%

[0081] The data show that 0.45% normalized surfactant in R-30 batts improved the mechanical property of recovery by nearly 2% after the aged hot/humid condition treatment. The surfactant modestly improved the sag properties in the ambient condition as well.

Example 6: Testing R-19 batts for Compressive Strength

[0082] R-19 fibrous batts are prepared as in Example 1. A package contains 5 bags of batts, each bag containing a plurality of batts. Packages are tested for Compressive Strength in the cross machine direction (CD) by measuring the width after compression to a standardized force of 1800 psi. The ambient results of N=8 measurements are shown in Figure 4 and Table 6, below:

[0083] Table 6: Compression of R-19 batts

% Surfactant	Mean Width, Compressed	Min	Std Dev	% Improvement, Compr. Width	% Improvement, Variability
0=control	17.85	16.00	1.44	--	--
0.09	19.08	17.63	0.69	6.9%	52.2%
0.27	19.83	18.50	0.97	11.1%	32.7%
0.44	20.80	20.63	0.17	16.5%	87.8%

[0084] The data show that the addition of surfactant in R-19 batts improved the mechanical property of compressive strength, with 0.44% normalized surfactant level improving compressive strength by at least 5%, at least 10% or even more than 15%. Interestingly, not only does compressive strength improve, but the variability of the data also improved a great deal; the standard deviation of the data points decreased nearly 10 fold. This is very important since, in at least the situation where compressive strength is important for stacking of packages, it is not the mean compression strength that matters, but the minimum. In stacks of packages, the weakest package will cause the stack to lean and possibly tumble.

Example 7: Surface tension with various surfactants

[0085] Several binder formulations were prepared with different types and amounts of binders as shown below in Table 7. Surface tension of the binder compositions were measured using a Surface Tensionmeter 6000 (manufactured by the SensaDyne Instrument Division of the Chem-Dyne Research Group). The instrument was calibrated with deionized water. Data was recorded every 5 seconds. After the system was stabilized and the testing had begun, the average value over a one-minute testing period was obtained for each sample. The results are set forth in Table 7.

[0086] Table 7: Surface tension of binder formulations with various surfactants

Binder Mixture (10% total solids)	Surfactant	% on total solids	Surface Tension (dyne/cm)
phenol/urea/formaldehyde (Control)	None	None	72.0
80:20 MD-CA w/5% SHP	None	None	77.7
80:20 MD-CA w/5% SHP	Stanfax ⁽¹⁾ (anionic)	0.1	46.0
		0.3	41.3
		0.5	41.9
80:20 MD-CA w/5% SHP	Surfynol 465 ⁽²⁾ (nonionic)	0.1	51.0
		0.3	49.4
		0.5	46.2
80:20 MD-CA w/5% SHP	Triton™ GR- PG70 ⁽³⁾	0.1	35.6
		0.3	31.3

	(anionic)	0.5	30.1
80:20 MD-CA w/5% SHP	Sodium Dodecyl-	0.1	60
	Sulfate	0.3	51.9
	(anionic)	0.5	50.8
80:20 MD-CA w/5% SHP	Triton™ CF-10 (nonionic)	0.1	39.1
		0.3	39.3
		0.5	40

(1) Stanfax – sodium lauryl sulfate

(2) Surfynol 465 – ethoxylated 2,4,7,9-tetramethyl 5 decyn-4,7-diol

(3) Triton™ GR-PG70 – 1,4-bis(2-ethylhexyl) sodium sulfosuccinate

(4) Triton™ CF-10 – poly(oxy-1,2-ethanediyl), alpha-(phenylmethyl)-omega-(1,1,3,3-tetramethylbutyl)phenoxy

** MD = maltodextrin, CA = citric acid, SHP = sodium hypophosphite

[0087] The results set forth in Table 7 show that all surfactants reduced the surface tension of the bio-based binder compared to both phenolic and carbohydrate-based control binders not having surfactants by an average of 40% plus/minus ~6% (95% CI). The magnitude of reduction ranged from about 17% for lowest concentration of sodium dodecyl sulfate surfactant to about 60% for the highest concentration of Triton GR-PG70. The two non-ionic surfactants produced surface tension reductions in the range of about 30-45% against the phenolic control and about 35-50% against the MD:CA control.

Example 8: Flexible Duct Media (FDM)

[0088] Flexible Duct Media (FDM) is made on a rotary fiberizing line and bisected to top and bottom portions, and rolled for packaging. Samples were made with and without 0.18% Surfynol, and targeting 6.0% LOI, making the calculated amount of surfactant in the fibrous product equal to 0.0106%. Samples are tested for recovery at an initial or ambient time, and again after an accelerated hot and humid (H/H) aging test. Individual data for top and bottom are averaged. The accelerated test exposed the FDM to 73°F and 92% relative humidity for 7 days. The results are shown in Table 8, below.

[0089] Table 8: H/H aged recovery data (inches) for FDM

	Ambient (inches)	73F/92RH @ 3 days, (inches)	73F/92RH @ 7 days, (inches)	Degradation at 7 days (%)
No Surfactant, control	2.38	2.14	2.04	13.96
0.18%Surfactant	2.31	2.23	2.11	8.78

[0090] The results show that 0.18% surfactant improved aged recovery. Recovery suffered almost 14% degradation without surfactant but less than 9% with surfactant.

[0091] The foregoing description of the various aspects and embodiments of the present invention has been presented for purposes of illustration and description. It is not intended to be exhaustive or all embodiments or to limit the invention to the specific aspects disclosed. Additional advantages and modifications will readily appear to those skilled in the art. Obvious modifications or variations are possible in light of the above teachings and such modifications and variations may well fall within the scope of the invention as determined by the appended claims when interpreted in accordance with the breadth to which they are fairly, legally and equitably entitled.

What is claimed is:

1. A method for manufacturing a fibrous insulation product having improved mechanical properties upon aging, the method comprising:
forming a fibrous product from a plurality of mineral fibers,
applying a thermosetting binder to the fibers of the fibrous product, the thermosetting binder including a polyhydroxyl compound and a polycarboxylic acid, the polyhydroxyl compound and the polycarboxylic acid being capable of forming crosslinks, and
applying a surfactant to the fibers of the fibrous product, the surfactant being applied to achieve a normalized concentration of about 0.01% to about 10% based on the dry weight of the fibrous product.
2. The method of claim 1, wherein the thermosetting binder is a natural binder comprising a carbohydrate-based polyhydroxyl compound and a polycarboxylic acid.
3. The method of claim 2, wherein the carbohydrate-based polyhydroxyl compound comprises a polysaccharide selected from a starch, a maltodextrin, a dextrin and a syrup.
4. The method of claim 2, wherein the carbohydrate-based polyhydroxyl compound has a dextrose equivalent (DE) from about 2 to about 20.
5. The method of claim 2, wherein the polycarboxylic acid is selected from a polyacrylic acid and an organic di- or tri- carboxylic acid.
6. The method of claim 2, wherein the surfactant is applied as a dispersion also containing the natural binder.
7. The method of claim 1, wherein the surfactant is applied at a normalized concentration from about 0.05 % to about 1%, based on the dry weight of the fibrous product.
8. The method of claim 1, wherein the surfactant is a non-ionic surfactant.
9. The method of claim 8, wherein the non-ionic surfactant is an ethoxylated polyalcohol.
10. The method of claim 9, wherein the non-ionic surfactant is selected from the Surfynol® series 420, 440, 465, and 485.
11. The method of claim 1, further comprising improving a mechanical property selected from recovery, sag, compressive strength, and tensile strength.
12. The method of claim 1, further comprising improving an aged mechanical property selected from recovery, sag, compressive strength, and tensile strength.

13. The method of claim 12, wherein recovery is improved by at least 2% compared to the same product made with without surfactant.

14. The method of claim 12, wherein sag is improved by at least 10% compared to the same product made with without surfactant.

15. The method of claim 12, wherein compressive strength is improved by at least 5% compared to the same product made with without surfactant.

16. The method of claim 1, wherein the surfactant is applied in combination with a silane coupling agent.

17. A method for improving the aged mechanical properties of a fibrous insulation product, the method comprising:

forming a fibrous product from a plurality of mineral fibers and a natural, thermosetting binder, the binder including a carbohydrate-based polyhydroxyl compound and a polycarboxylic acid, the polyhydroxyl compound and the polycarboxylic acid being capable of forming crosslinks, and

applying a surfactant to the fibers of the fibrous product, the surfactant being applied to achieve a normalized concentration of about 0.05% to about 5% based on the dry weight of the fibrous product; wherein at least one aged mechanical property is improved by at least 2% compared to fibrous product not containing the surfactant.

18. The method of claim 17, wherein the aged mechanical property is selected from recovery, sag, compressive strength, and tensile strength.

19. The method of claim 17, wherein the surfactant is a non-ionic ethoxylated polyalcohol.

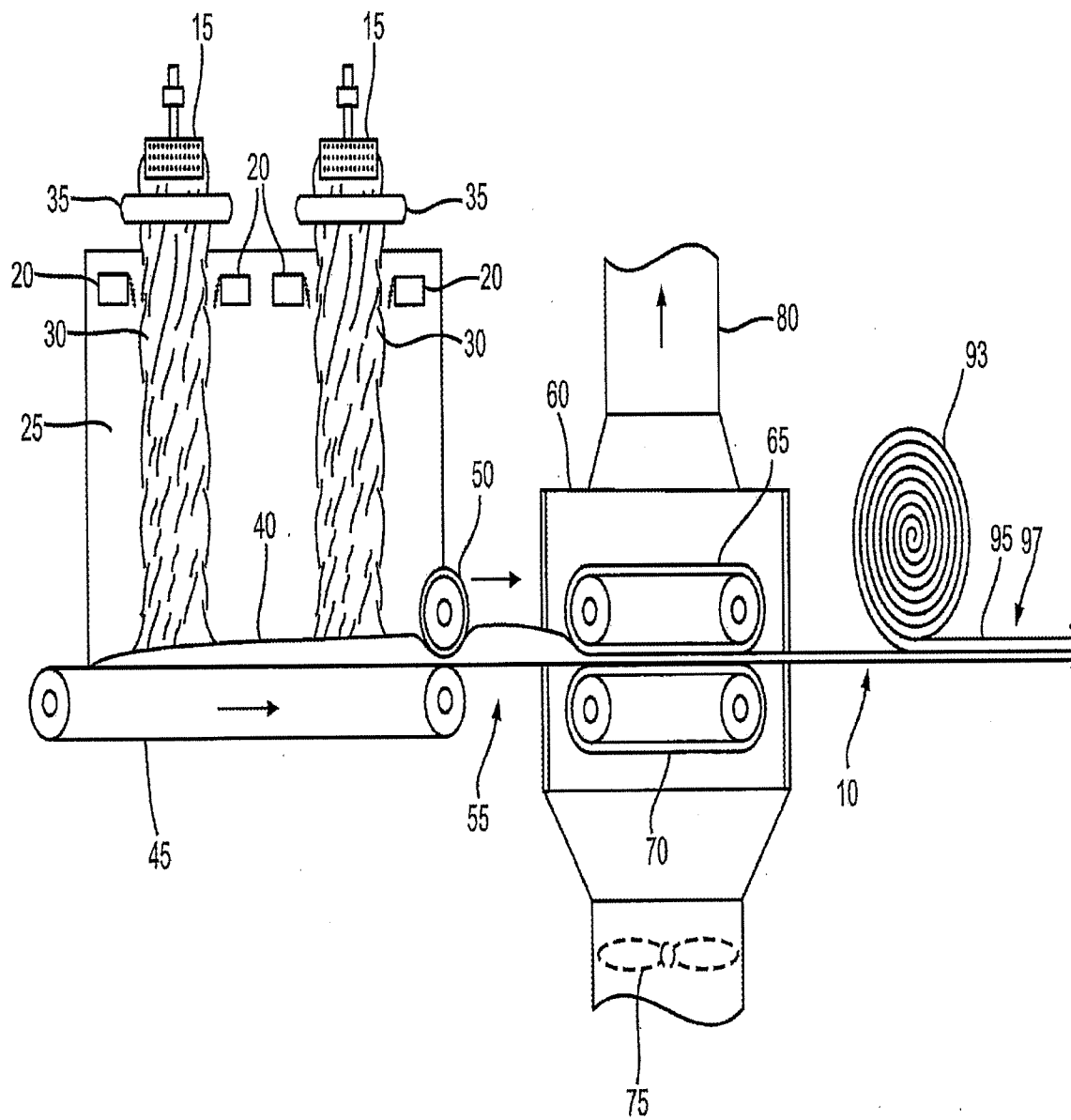
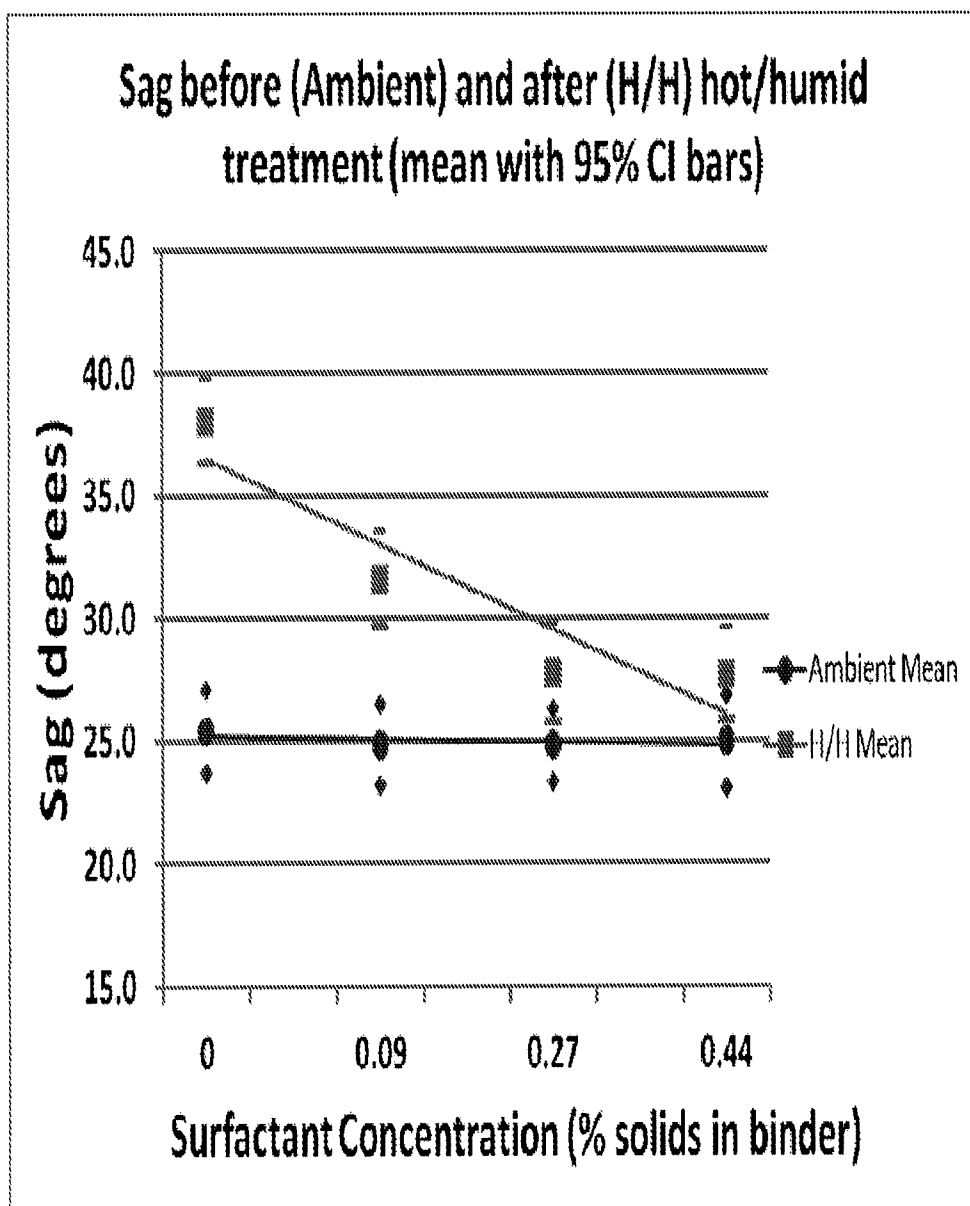
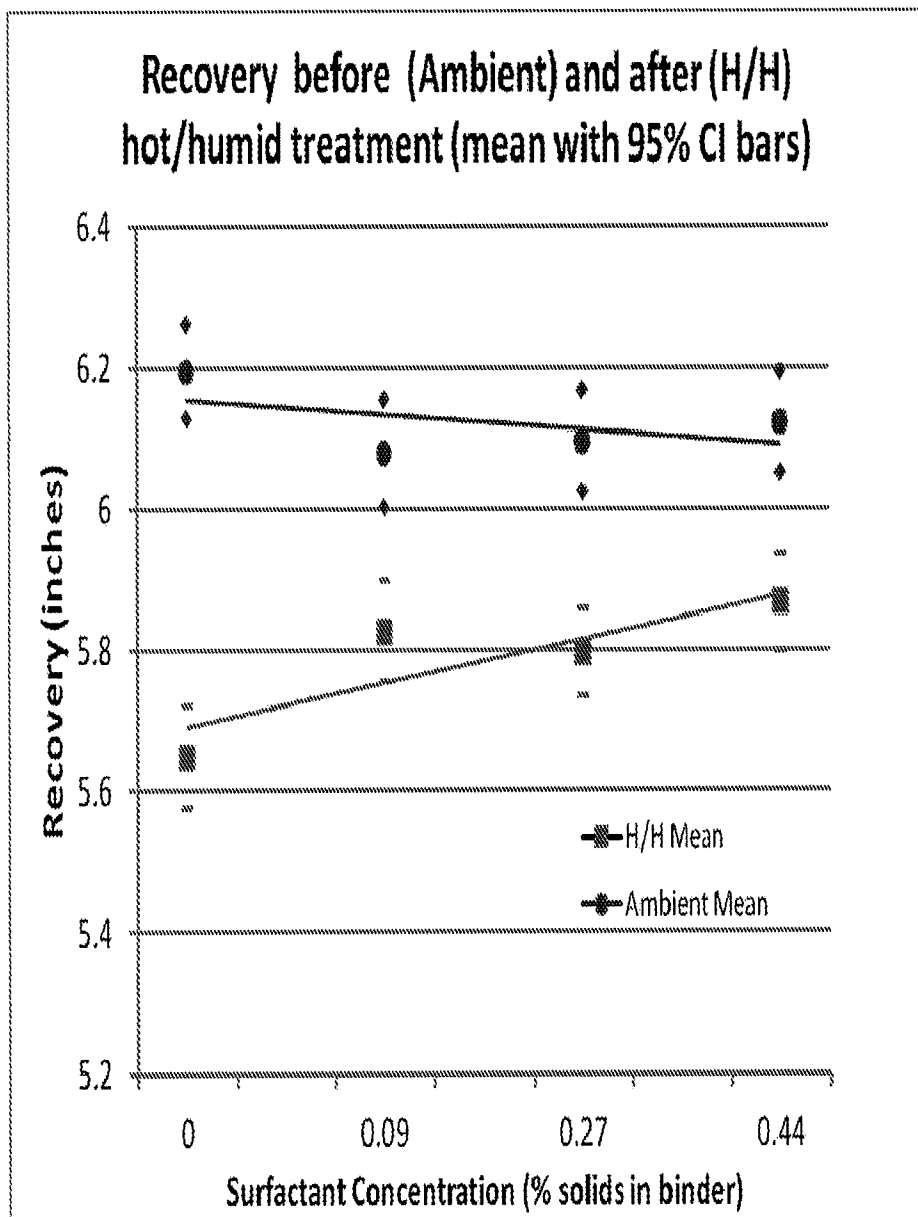
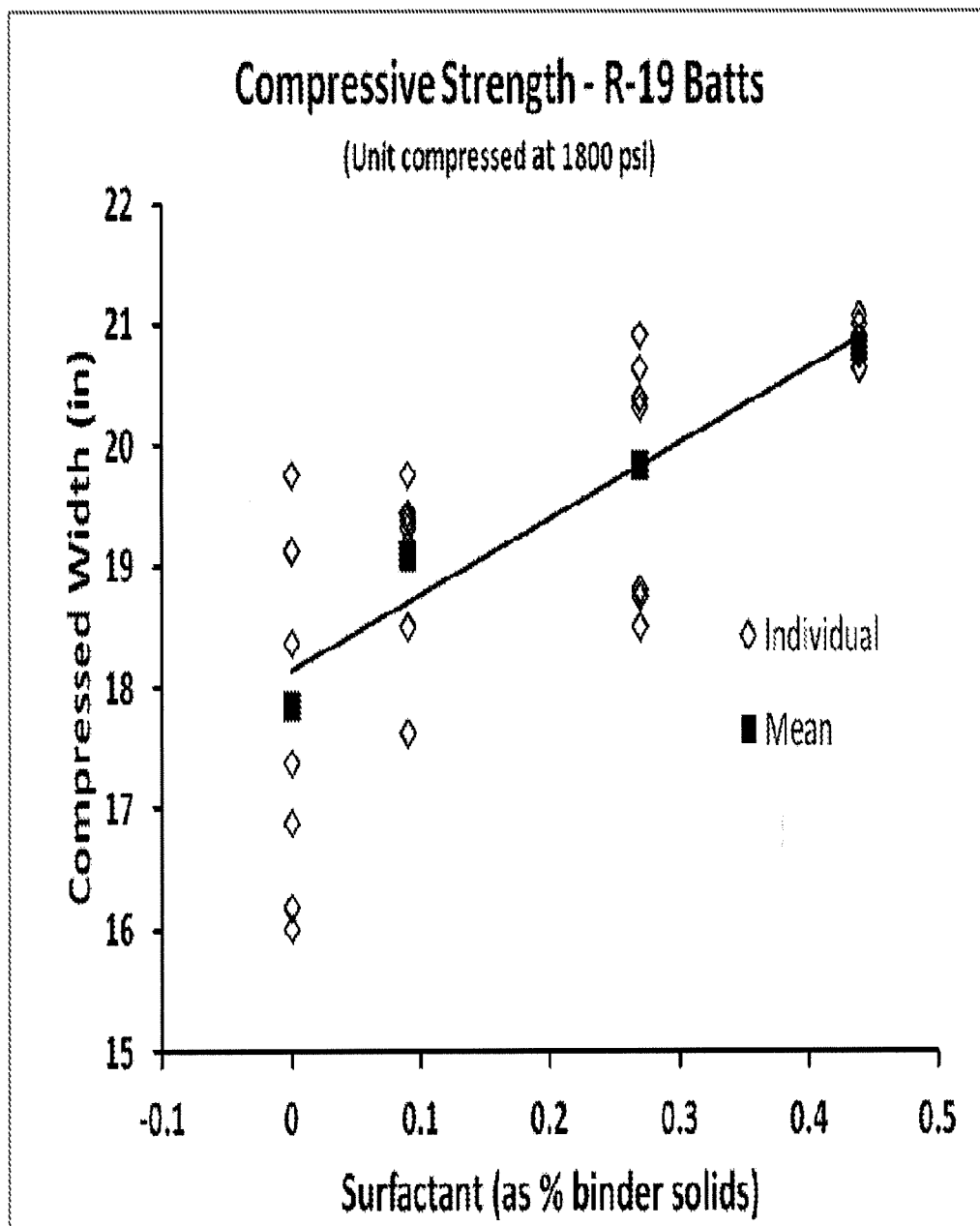


Fig. 1

**Fig. 2**

**Fig. 3**

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**Fig. 4**

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2013/039384

A. CLASSIFICATION OF SUBJECT MATTER

IPC(8) - D04H 1/64 (2013.01)

USPC - 442/172

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC(8) - C03C 13/00, 13/06, 25/00, 25/10; D04H 1/00, 1/40, 1/4218, 1/58, 1/64 (2013.01)
USPC - 162/141, 145; 428/34.5, 292.1; 442/172

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
CPC - C03C 13/00, 13/06, 25/00, 25/10; D04H 1/00, 1/40, 1/4218, 1/58, 1/64 (2013.01)

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
Orbit, Google Patents, Google Scholar

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 2011/0086567 A1 (HAWKINS et al) 14 April 2011 (14.04.2011) entire document	1-19
Y	US 5,744,229 A (GLEASON et al) 28 April 1998 (28.04.1998) entire document	1-19
Y	US 2009/0156724 A1 (ESPIARD et al) 18 June 2009 (18.06.2009) entire document	13, 17-19
Y	US 3,243,340 A (CADOTTE) 29 March 1966 (29.03.1966) entire document	14
Y	US 2009/0054580 A1 (JOACHIM et al) 26 February 2009 (26.02.2009) entire document	15

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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

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Date of the actual completion of the international search

24 September 2013

Date of mailing of the international search report

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