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(57) Abstract: A stable suspension used for the production of temperature regulating fabrics. The suspension preferably comprises microcapsules comprising at least one phase-change material. A method for manufacturing the stable suspension that includes providing microcapsules containing a phase-change material, providing a solvent capable of dissolving a fabric-forming component selected from the group consisting of at least one of said polymer and precursors thereof, and mixing said solvent and said microcapsules to form said first suspension.



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STABLE SUSPENSIONS CONTAINING MICROCAPSULES AND METHODS FOR THE PREPARATION THEREOF

PRIORITY CLAIM AND CROSS REFERENCE TO RELATED APPLICATIONS

[001] This application claims the benefit of Israeli Patent Application No. 176693 filed on July 4, 2006, the disclosure of which is incorporated herein by reference in its entirety.

FIELD OF THE INVENTION

[002] This invention relates generally to temperature regulating fabrics. More particularly, the present invention relates to a stable suspension used for the production of such fabric, which suspension comprises microcapsules comprising at least one phase-change material.

BACKGROUND OF THE INVENTION

[003] The human body maintains its temperature at a constant level, by use of the sweating process for cooling and body hair as an isolation medium.

[004] However, the natural human body temperature adjusting capabilities are limited, and are disturbed by clothing.

[005] Temperature-regulating material, such as phase-change material, when placed near the body can help regulate skin and microclimate temperatures. Phase-change material with a phase-changing temperature in a desired range, absorbs energy from the skin as the body temperature rises, and releases heat to the body as the body cools down. This results in less fluctuation in the human skin and microclimate temperatures, a greater feeling of comfort, less sweating, and lower microclimate relative humidity.

[006] Phase-change material can also function as an energy saver, e.g., if a room is furnished with an element containing phase-change material, such as paints, carpets, furniture finish, furniture fabrics or furniture coating, the element absorbs excessive heat during the warmer hours, and releases it during cooler hours. In a similar manner, it may also be used in building and construction materials such as insulation, roofing, wallboards, wallcoverings, ceiling materials, floor and floor covering materials, etc. Furthermore, phase-change materials may also be used in numerous other markets where temperature regulation, or temperature buffering, may

have a benefit, such as: healthcare, apparel, electronics, transportation, shipping, cosmetics/personal care, food and beverage packaging, appliances, disposables and more.

[007] In general, a temperature-regulating material may comprise any substance (or mixture of substances) that has the capability of absorbing or releasing thermal energy to reduce or eliminate heat flow at or within a temperature stabilizing range. The temperature stabilizing range may comprise a particular transition temperature or range of transition temperatures. A phase-change material, if properly located in the final product, is capable of inhibiting a flow of thermal energy during a time when the phase-change material is absorbing or releasing heat, typically as the phase-change material undergoes a transition between two states (e.g., liquid and solid states, liquid and gaseous states, solid and gaseous states, or two solid states). This action is typically transient, e.g., occurs until a latent heat of the phase-change material is absorbed or released during a heating or cooling process. Thermal energy may be stored or removed from the phase-change material, and the phase-change material typically can be effectively recharged by a hot or cold source.

[008] Phase-change material normally is encapsulated. Encapsulation is desired for some of the following reasons: protection against leakage of the phase-change material after the phase-change to a liquid state; and protection of phase-change material from contamination, increased durability; product feel, etc.

[009] For some applications, e.g., clothing, it is preferred that the capsules be in the micrometer / nanometer size range. In these size ranges, the phase-change material capsules can be incorporated into a product without change in the appearance, texture or production process of the product. Capsules in the micrometer / nanometer size range are referred to here as microcapsules. Phase-change material encapsulated with microcapsules is referred to as microencapsulated phase-change material (mPCM)

[0010] Microencapsulated phase-change material can be introduced into commercial products in various ways. According to some industrial practices, mPCM, typically in combination with binders, and possibly other components, is coated on commercial products. Coating may use known methods, such as knife-over-roll coating, roll coating, slot coating, screenprinting, foam

coating, laminating, exhausting, spraying, padding, extrusions, embossing or flocking e.g. as described in U.S patents; 5,366,801; 6,207,738; 6,217,993; 6,503,976; 6,514,362; and 6,660,667, the relevant teachings of which are incorporated here by reference.

[0011] Many commercial products are made of fibers. Microencapsulated phase-change material can be coated on fibers prior to conversion to the final commercial product or after it. Alternatively, mPCM may be incorporated into the fibers in the process of their manufacture.

[0012] Conventionally, two processes are used to manufacture synthetic fibers: a solution spinning process and a melt spinning process. The solution spinning process is generally used to form acrylic or regenerated cellulosic fibers, while the melt spinning process is generally used to form nylon fibers, polyester fibers, polypropylene fibers, and other similar type fibers. The solution spinning process is divided into two main spinning techniques; dry spinning and wet spinning. In the wet spinning process the spinnerets are submerged in or held very close to a chemical bath and as the filaments emerge they contact the chemical bath and precipitate from solution and solidify. In the dry spinning process instead of precipitating the polymer by dilution or chemical reaction, solidification is achieved by evaporating the solvent in a stream of air or inert gas which can be heated or cooled.

[0013] Several methods have been developed for the incorporation of phase-change material and microencapsulated phase-change material into fibers, as described for example in US Patents 6,855,422; 6,689,466; and 4,756,958 and US Patent Applications 20050208300; 20040126555; 20020054964, and in *Acrylic Fibers* by R. Cox in *Synthetic Fibers: Nylon, Polyester, Acrylic, Polyolefins*, Woodhead Publishing ISBN 1 85573 588 1, the relevant teachings of which are incorporated here by reference. Many of these methods suffer from a common difficulty related to the dispersion of the microencapsulated phase-change material (mPCM). During the process of fiber manufacture, the incorporated microcapsules tend to form agglomerates of larger particles. This agglomeration may lead to manufacturing and yield problems and to the production of unattractive mPCM-containing fibers. Particularly affected are the physical properties of the fiber such as strength, denier variation, thick and thin spots, etc. If microcapsules agglomerate, production problems such as filter blockage, deposition on pipe

walls, pressure and flow variations and spinneret hole blockage may occur. This further causes changes in fiber denier and or production line stoppage.

[0014] In some of the manufacturing processes, a suspension of mPCM in a solution is produced and then mixed with a solution of a polymer or of a polymer-precursor (e.g. monomer) for spinning. The manufacturing process is simplified if a suspension of mPCM is formed in a way that is stable enough to enable storage prior to mixing with the polymer (or precursor) for a prolonged period with no substantial agglomeration or phase separation. Furthermore, after mixing, the mPCM should be evenly dispersed in the mixture to enable the production of fibers with desired distribution of mPCM. Achieving such desired stable suspensions and even dispersion is difficult in many cases, e.g. due to the high ionic strength of the solution. In many cases "creaming" is observed. This term is used to describe the formation of two layers, with the microcapsules presiding in the top layer. If creaming occurs in pipe work, it forms a skin or coating on the pipe work which is very difficult to remove.

SUMMARY OF THE INVENTION

[0015] Thus according to one aspect of the present invention, there is now provided a stable first suspension for the production of a temperature-regulating, polymer-containing fabric, said suspension comprising a solvent and a plurality of microcapsules containing at least one phase-change material, wherein said microcapsules are to be incorporated in said polymer-containing fabric, and wherein said stable first suspension is characterized in that said solvent is capable of dissolving a fabric-forming component selected from the group consisting of at least one of said polymer and precursors thereof; and said suspension is stable for at least about 20 hours.

[0016] In preferred embodiments of the present invention, not only is the suspension stable for at least about 20 hours, meaning that no significant aggregation is noted, but the microcapsules in the suspension are also stable to degradation. Such degradation may lead to undesired leakage of phase-change material, if in liquid form.

[0017] Thus, in preferred embodiments, at least about 95% of the microcapsules in the suspension stay intact in the suspension for at least about 20 hours. According to an especially preferred embodiment, at least about 98% of the microcapsules in the suspension stay intact in

the suspension for at least about 20 hours. In the most preferred embodiments, at least about 98% of the microcapsules in the suspension stay intact in the suspension for at least about 40 hours.

[0018] In other preferred embodiments of the present invention said fabric is formed from at least one type of polymeric fiber.

[0019] Preferably said polymer is selected from the group consisting of acrylonitrile-based polymers, cellulose-based polymers, polyester-based polymers, polyamide-based polymers and polyolefin based polymers.

[0020] In some preferred embodiments of the present invention said solvent is an aqueous solution.

[0021] Preferably said aqueous solution comprises at least one further component selected from the group consisting of sodium bases, sodium thiocyanate, zinc chloride, n-methyl morpholine oxide, ammonia, copper sulfate nitric acid, acetone, dimethyl formamide (DMF) and n-methyl pyrrolidone (NMP).

[0022] In yet other preferred embodiments of the present invention said first suspension further comprises at least one further component selected from the group consisting of a defoaming agent, a flow-control agent, a wetting agent, a dispersing agent and surfactant.

[0023] Preferably said aqueous solutions comprise at least one further component selected from the group consisting of sodium bases, sodium thiocyanate, zinc chloride, n-methyl morpholine oxide, ammonia, copper sulfate, nitric acid, acetone, DMF and NMP.

[0024] In other aspects of the present invention said microcapsules comprise a shell and a core and said shell is formed from at least one component selected from the group consisting of acrylic acid and derivatives thereof, methacrylic acid and derivatives thereof, formaldehyde, isocyanate, urea, carboxylic acid derivatives, silica precursor and gelatin.

[0025] The microcapsules may comprise a shell and a core, wherein said core comprises a phase-change material selected from the group consisting of octadecane, straight-chain hydrocarbons with 15 to 25 carbon atoms, branched-chain hydrocarbons with 15 to 25 carbon atoms.

[0026] According to another embodiment of the invention the at least one phase-change material has a melting point in a range between about 20°C and about 50°C. According to still another embodiment of the invention the at least one phase-change material has a melting enthalpy in a range between about 80 J/g and about 400 J/g.

[0027] In preferred embodiments of the present invention said microcapsules comprise a shell and a core and the weight ratio between said shell and core is in the range between about 0.5:9.5 and about 4:6.

[0028] The weight ratio may also be in a range between about 1:9 to about 3:7.

[0029] The microcapsules may form between about 5 and about 40 percent by weight of said suspension.

[0030] The microcapsules may form between about 10 and about 30 percent by weight of said suspension.

[0031] The microcapsules may have a variety of regular or irregular shapes (e.g., spherical, ellipsoidal, and so forth) and variety of sizes. The individual microcapsules may have the same or different shapes or sizes. According to an embodiment of the invention, the microcapsules have a maximum linear dimension ranging between about 0.1 and about 20 micron. Preferably the microcapsules have a maximum linear dimension ranging between about 0.3 and about 2 micron.

[0032] In another aspect of the present invention, the polymer is acrylonitrile-based, the solvent of the stable suspension comprises water and sodium thiocyanate, the concentration of the sodium thiocyanate ranges between about 40 and about 60 percent by weight of the solvent and

the microcapsules form between about 5 and about 30 percent by weight of said suspension. According to a related embodiment, the polymer is acrylonitrile-based, the solvent of the stable suspension comprises water and sodium thiocyanate, the concentration of the sodium thiocyanate ranges between about 45 and about 55 percent by weight of the solution and the microcapsules form between about 8 and about 20 percent by weight of said suspension and the suspension is kept at an ambient temperature.

[0033] In other aspects of the present invention said polymer is cellulose-based, wherein said solvent comprises water and sodium hydroxide, wherein the concentration of the sodium hydroxide ranges between about 1 percent and about 5 percent and wherein said microcapsules form between about 5 and about 30 percent of said suspension.

[0034] In another aspect of the present invention there is provided a second suspension formed from the first suspension as defined above, in combination with at least one polymeric fiber forming component selected from the group consisting of a polymer and a precursor thereof.

[0035] Said microcapsules may be evenly dispersed throughout said second suspension.

[0036] According to another aspect, the second suspension contains microcapsules incorporating at least one functional compound, said functionality being selected from the group consisting of fire retardation, bioactivity, antimicrobial activity, odor resistance, UV absorption, moisture management and resistance to water, grease, dirt and/or stain.

[0037] According to various other embodiments, such microcapsules are present in said stable suspension prior to said combining, introduced with the combined polymer or precursor, introduced after said combining or any combination of those options. According to a preferred embodiment, all types of microcapsules are evenly dispersed in the second suspension.

[0038] In cases where the stable suspension is used for the production of temperature-regulating, polymer-containing fabric, the solvent is preferably selected so that it is capable of dissolving the polymer and/or a precursor for its formation. According to an embodiment of the invention, the solvent is an aqueous solution. For example, an aqueous solution of sodium thiocyanate is a

suitable solution in case the polymer is acrylonitrile based while an aqueous solution of sodium hydroxide is a suitable solution if the polymer is cellulose based. According to an embodiment of the invention the solvent is provided at an ambient temperature. The solvent and a plurality of microcapsules are mixed to form the stable suspension. According to an embodiment of the invention, the solvent is added to the microcapsules while being mixed with a low-shear mixer.

[0039] In another aspect of the present invention there is provided a method for manufacturing of said first suspension as defined hereinbefore, comprising the steps of providing microcapsules containing a phase-change material; providing a solvent capable of dissolving a fabric-forming component selected from the group consisting of at least one of said polymer and precursors thereof; and mixing said solvent and said microcapsules to form said first suspension.

[0040] In one embodiment, said solvent is provided at a temperature in the range of between about ambient and 120 °C.

[0041] In one embodiment, said first suspension is kept at a temperature in the range of between about ambient and 120 °C.

[0042] In one embodiment, said first suspension is kept under low-shear mixing.

[0043] Said microcapsules may be provided in a vessel and said solvent is added to said microcapsules in said vessel to form said first suspension. Said solvent may be an aqueous solvent.

[0044] Said microcapsules may be provided as a third suspension of microcapsules in a fluid and said third suspension may be in an aqueous medium.

[0045] The weight ratio between the microcapsules and the water in the third suspension may be in a range between about 30 and 80 percent. That third suspension is generated in any known way, e.g. by mixing microcapsules with water or with an aqueous solution. In some case, a suspension is generated in the process of manufacturing the phase-change material-comprising

microcapsules. In such cases, that suspension is used as the third suspension, as such, or after some modification.

[0046] In one aspect of the present invention, the viscosity of said third suspension is in the range of between about 100 and about 3000 cps.

[0047] In yet other aspects of the present invention the viscosity of said third suspension is in the range of between about 1000 and about 2000 cps.

[0048] Preferably said third suspension is provided at a temperature in the range of between about ambient and 120 °C.

[0049] In other embodiments of this aspect of the present invention said method further comprises the step of adding at least one further component selected from the group consisting of a defoaming agent, wetting agent a flow-control agent, a dispersing agent and a surfactant to said first suspension.

[0050] In yet another preferred embodiment of the present invention there is provided a method for the manufacturing of said second suspension as defined hereinabove comprising the steps of providing at least one fabric-forming component selected from the group consisting of a polymer and a precursor thereof and combining said component with said first suspension to form said second suspension.

[0051] The polymer and/or precursor is provided in any form, for example in a solution. Any solvent capable of dissolving the polymer and/or precursor is suitable for such polymer and/or precursor solution. According to an embodiment of the invention that solvent is composed of components used as solvent in the formation of the first suspension. According to another embodiment, the solvent of the polymer and/or precursor solution is similar in composition to the solvent in the first suspension, e.g. both are aqueous solutions of sodium thiocyanate when the polymer is acrylonitrile based and aqueous solutions or sodium hydroxide, when the polymer is cellulose based. Combining could use known method, such as mixing. According to an embodiment of the invention, the microcapsules are evenly dispersed in the second suspension.

[0052] In this aspect of the present invention said method further comprises wet spinning or dry spinning of said second suspension.

[0053] The invention is also directed to a fiber whenever manufactured according to one of said spinning steps.

[0054] In preferred embodiments of the present invention said microcapsules are evenly dispersed throughout said fiber.

[0055] In this aspect of the present invention said fiber is preferably combined with other fibers to form a fabric.

[0056] In other preferred embodiments of the present invention there is provided a commercial product comprising a fiber produced by said method.

BRIEF DESCRIPTION OF THE DRAWINGS

[0057] FIG. 1 is a graphic representation of particle size distribution of a freshly formed stable suspension of microcapsules produced according to Example 3; and

[0058] FIG. 2 is a graphic representation of particle size distribution of a stable suspension of microcapsules produced according to Example 3 after 24 hours.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0059] As used herein the term “stable suspension” can refer, for example, to a suspension wherein a plurality of microcapsules comprising phase-change material is suspended in a solvent and wherein the majority of said microcapsules are evenly dispersed. The term can also refer to that suspension, wherein the majority of the microcapsules are not substantially agglomerated. The term can also refer to that suspension, wherein the majority of the microcapsules do not float on top of the solvent nor sink to its bottom. The term can also refer to that suspension, wherein no creaming takes place.

[0060] As used herein the term “polymer precursor” refers, for example, to compounds that are converted to a polymer by polymerization, optionally with other compounds. Monomers are examples of polymer precursors.

[0061] As used herein the term “evenly dispersed” refers, for example, to a state where the majority of the microcapsules are not agglomerated.

[0062] As used herein the term “solvent capable of dissolving” refers, for example, to a solvent wherein the polymer or its precursor is soluble to the extent that a solution containing at least 1 weight percent of the solute can be prepared.

[0063] As used herein, the term “latent heat” refers, for example, to the quantity of energy absorbed or released by a substance undergoing a change of state.

[0064] As used herein, the term “phase-change material” refers, for example, to a material that has the capability of absorbing or releasing energy to adjust heat transfer at or within a temperature stabilizing range. A temperature stabilizing range can include a specific transition temperature or a range of transition temperatures. In some instances, a phase-change material can be capable of inhibiting heat transfer during a period of time when the phase-change material is absorbing or releasing heat, typically as the phase-change material undergoes a transition between two states. This action is typically transient and will occur until a latent heat of the phase-change material is absorbed or released during a heating or cooling process. Heat can be stored or removed from a phase-change material, and the phase-change material typically can be effectively recharged by a source of heat or cold. For certain implementations, a phase-change material can be a mixture of two or more materials. By selecting two or more different materials and forming a mixture, a temperature stabilizing range can be adjusted for any desired application. The resulting mixture can exhibit two or more different transition temperatures or a single modified transition temperature when incorporated in accordance with the following articles.

“A Review on Phase-change Energy Storage: Materials and Applications” by Farid, M.M., et. al in *Energy Conversion and Management* 45, (2004) 1597-1615.

“Review on Thermal Energy Storage with Phase-change: Materials, Heat Transfer Analysis and Applications” by Zalba, B., et.al in *Applied Thermal Engineering* 23 (2003), 251-283.

“Actual Problems in Using Phase-Change Materials to Store Solar Energy” by Kenisarin, M., et.al , Paper presented at the NATO Advanced Study Institute Summer School on Thermal Energy Storage for Sustainable Energy Consumption (TESSEC), Cesme, Izmir, Turkey, June, 2005.

[0065] Phase-change materials that can benefit from stabilization in accordance with various embodiments of the invention include a variety of organic substances. Exemplary phase-change materials include, by way of example and not by limitation, hydrocarbons (e.g., straight chain alkanes or paraffinic hydrocarbons, branched-chain alkanes, unsaturated hydrocarbons, halogenated hydrocarbons, and alicyclic hydrocarbons), fatty acids, fatty acid esters, dibasic acids, dibasic esters, 1-halides, primary alcohols, aromatic compounds, anhydrides (e.g., stearic anhydride), ethylene carbonate, glycols, polyhydric alcohols (e.g., 2,2-dimethyl-1, 3-propanediol, 2-hydroxymethyl-2-methyl-1, 3-propanediol, polyethylene glycol, pentaerythritol, dipentaerythritol, pentaglycerine), polymers, polyglycols, metals, and mixtures thereof.

[0066] The selection of the phase-change material can be dependent upon a latent heat and a transition temperature of the phase-change material. A latent heat of the phase-change material typically correlates with its ability to reduce or eliminate heat transfer. In some instances, the phase-change material can have a latent heat that is at least about 40 J/g, such as at least about 50 J/g, at least about 60 J/g, at least about 70 J/g, preferably at least about 80 J/g, especially preferred at least about 90 J/g, and most preferred at least about 100 J/g. Thus, for example, the phase-change material can have a latent heat ranging from about 40 J/g to about 400 J/g, preferably from about 60 J/g to about 400 J/g, especially preferred from about 80 J/g to about 400 J/g, and most preferred from about 100 J/g to about 400 J/g. A transition temperature of the phase-change material typically correlates with a desired temperature or a desired range of temperatures that can be maintained by the phase-change material. In some instance, the phase-

change material can have a transition temperature ranging from about $-10\text{ }^{\circ}\text{C}$ to about $110\text{ }^{\circ}\text{C}$, such as from about $0\text{ }^{\circ}\text{C}$ to about $100\text{ }^{\circ}\text{C}$, from about $0\text{ }^{\circ}\text{C}$ to about $50\text{ }^{\circ}\text{C}$, from about $10\text{ }^{\circ}\text{C}$ to about $50\text{ }^{\circ}\text{C}$, preferably from about $15\text{ }^{\circ}\text{C}$ to about $45\text{ }^{\circ}\text{C}$, especially preferred from about $22\text{ }^{\circ}\text{C}$ to about $40\text{ }^{\circ}\text{C}$, and most preferred from $22\text{ }^{\circ}\text{C}$ to about $28\text{ }^{\circ}\text{C}$. The selection of the phase-change material can be dependent upon other considerations, such as its reactivity or lack of reactivity with a material forming the shells, its resistance to degradation under ambient or processing conditions, its biodegradability, and its toxicity.

[0067] Table 1 provides a list of exemplary paraffinic hydrocarbons that may be used as the phase-change material stabilized in the accordance with various embodiments of the invention.

TABLE 1

Paraffinic Hydrocarbon	No. of Carbon Atoms	Melting Point $^{\circ}\text{C}$.
n-Octacosane	28	61.4
n-Heptacosane	27	59.0
n-Hexacosane	26	56.4
n-Pentacosane	25	53.7
n-Tetracosane	24	50.9
n-Tricosane	23	47.6
n-Docosane	22	44.4
n-Heneicosane	21	40.5
n-Eicosane	20	36.8
n-Nonadecane	19	32.1
n-Octadecane	18	28.2
n-Heptadecane	17	22.0
n-Hexadecane	16	18.2
n-Pentadecane	15	10.0
n-Tetradecane	14	5.9
n-Tridecane	13	-5.5

[0068] A phase-change material can be a mixture of two or more substances (e.g., two or more of the exemplary phase-change materials discussed above). By selecting two or more different substances (e.g., two different paraffinic hydrocarbons) and forming a mixture thereof, a temperature stabilizing range can be adjusted over a wide range for any desired application.

According to some embodiments of the invention, a phase-change material may comprise a copolymer of two or more substances (e.g., two or more of the exemplary phase-change materials discussed above).

[0069] The selection of a phase-change material will typically be dependent upon a desired transition temperature or a desired application of a resulting multi-component fiber. For example, a phase-change material having a transition temperature near room temperature may be desirable for applications in which the resulting multi-component fiber is incorporated into apparel designed to maintain a comfortable temperature for a user.

[0070] According to some embodiments of the invention, particularly useful phase-change materials include paraffinic hydrocarbons having between 10 to 44 carbon atoms (i.e., C₁₀ –C₄₄ paraffinic hydrocarbons). Table 1 provides a list of exemplary C₁₃ –C₂₈ paraffinic hydrocarbons that may be used as the phase-change material in the silica capsules described herein. The number of carbon atoms of a paraffinic hydrocarbon typically correlates with its melting point. For example, n-Octacosane, which contains twenty-eight straight chain carbon atoms per molecule, has a melting point of 61.4°C. By comparison, n-Tridecane, which contains thirteen straight chain carbon atoms per molecule, has a melting point of -5.5°C. According to an embodiment of the invention, n-Octadecane, which contains eighteen straight chain carbon atoms per molecule and has a melting point of 28.2°C, is particularly desirable for clothing applications.

[0071] Other useful phase-change materials include polymeric phase-change materials having transition temperatures suitable for a desired application of the multi-component fiber (e.g., from about 22°C to about 40°C for clothing applications). A polymeric phase-change material may comprise a polymer (or mixture of polymers) having a variety of chain structures that include one or more types of monomer units. In particular, polymeric phase-change materials may include linear polymers, branched polymers (e.g., star branched polymers, comb branched polymers, or dendritic branched polymers), or mixtures thereof. A polymeric phase-change material may comprise a homopolymer, a copolymer (e.g., terpolymer, statistical copolymer, random copolymer, alternating copolymer, periodic copolymer, block copolymer, radial

copolymer, or graft copolymer), or a mixture thereof. As one of ordinary skill in the art will understand, the reactivity and functionality of a polymer may be altered by addition of a functional group such as, for example, amine, amide, carboxyl, hydroxyl, ester, ether, epoxide, anhydride, isocyanate, silane, ketone, and aldehyde. Also, a polymer comprising a polymeric phase-change material may be capable of crosslinking; entanglement; or hydrogen bonding in order to increase its toughness or its resistance to heat, moisture, or chemicals.

[0072] According to some embodiments of the invention, a polymeric phase-change material may be desired which has a higher molecular weight, larger molecular size, or higher viscosity relative to a non-polymeric phase-change material (e.g., paraffinic hydrocarbons). As a result of this larger molecular size or higher viscosity, a polymeric phase-change material may exhibit a lesser tendency to leak from its shell, for example, the larger molecular size or higher viscosity may prevent the polymeric phase-change material from flowing through a sheath member or seal member forming the exterior of the shell.

[0073] For example, polyethylene glycols may be used as the phase-change material in some embodiments of the invention. The average molecular weight of a polyethylene glycol typically correlates with its melting point. For instance, a polyethylene glycol having an average molecular weight range of 570 to 630 (e.g., Carbowax 600) will have a melting point of 20°C to 25°C. Other polyethylene glycols that may be useful at other temperature stabilizing ranges include Carbowax 400 (melting point of 4°C to 8°C.), Carbowax 1500 (melting point of 44°C. to 48°C), and Carbowax 6000 (melting point of 56 °C to 63 °C). Polyethylene oxides having a melting point in the range of 60°C to 65°C may also be used as phase-change materials in some embodiments of the invention. Further desirable phase-change materials include polyesters having a melting point in the range of 0°C to 40 °C, that may be formed, for example, by polycondensation of glycols (or their derivatives) with diacids (or their derivatives). Table 2 sets forth melting points of exemplary polyesters that may be formed with various combinations of glycols and diacids.

TABLE 2

Glycol	Diacid	Melting Point of Polyester (° C.)
Ethylene glycol	Carbonic	39
Ethylene glycol	Pimelic	25
Ethylene glycol	Diglycolic	17-20
Ethylene glycol	Thiodivaleric	25-28
1,2-Propylene glycol	Diglycolic	17
Propylene glycol	Malonic	33
Propylene glycol	Glutaric	35-39
Propylene glycol	Diglycolic	29-32
Propylene glycol	Pimelic	37
1,3-butanediol	Sulphenyl divaleric	32
1,3-butanediol	Diphenic	36
1,3-butanediol	Diphenyl	38
	methane-m,m'-diacid	
1,3-butanediol	trans-H,H-terephthalic acid	18
Butanediol	Glutaric	36-38
Butanediol	Pimelic	38-41
Butanediol	Azelaic	37-39
Butanediol	Thiodivaleric	37
Butanediol	Phthalic	17
Butanediol	Diphenic	34
Neopentyl glycol	Adipic	37
Neopentyl glycol	Suberic	17
Neopentyl glycol	Sebacic	26
Pentanediol	Succinic	32
Pentanediol	Glutaric	22

TABLE 2-continued

Glycol	Diacid	Melting Point of Polyester (° C.)
Pentanediol	Adipic	36
Pentanediol	Pimelic	39
Pentanediol	para-phenyl diacetic acid	33
Pentanediol	Diglycolic	33
Hexanediol	Glutaric	28-34
Hexanediol	4-Octenedioate	20
Heptanediol	Oxalic	31
Octanediol	4-Octenedioate	39
Nonanediol	meta-phenylene diglycolic	35
Decanediol	Malonic	29-34
Decanediol	Isophthalic	34-36
Decanediol	meso-tartaric	33
Diethylene glycol	Oxalic	10
Diethylene glycol	Suberic	28-35
Diethylene glycol	Sebacic	36-44
Diethylene glycol	Phthalic	11
Diethylene glycol	trans-H,H-terephthalic acid	25
Triethylene glycol	Sebacic	28
Triethylene glycol	Sulphonyl divaleric	24
Triethylene glycol	Phthalic	10
Triethylene glycol	Diphenic	38
para-dihydroxy-methyl benzene	Malonic	36
meta-dihydroxy-methyl benzene	Sebacic	27
meta-dihydroxy-methyl benzene	Diglycolic	35

[0074] According to some embodiments of the present invention, a polymeric phase-change material having a desired transition temperature may be formed by reacting a phase-change material (e.g., an exemplary phase-change material discussed above) with a polymer (or mixture of polymers). Thus, for example, n-octadecylic acid (i.e., stearic acid) may be reacted or esterified with polyvinyl alcohol to yield polyvinyl stearate, or dodecanoic acid (i.e., lauric acid) may be reacted or esterified with polyvinyl alcohol to yield polyvinyl laurate. Various combinations of phase-change materials (e.g., phase-change materials with one or more functional groups such as amine, carboxyl, hydroxyl, epoxy, silane, sulfuric, and so forth) and

polymers may be reacted to yield polymeric phase-change materials having desired transition temperatures.

[0075] A phase-change material can comprise a mixture of two or more substances (e.g., two or more of the exemplary phase-change materials discussed above). By selecting two or more different substances (e.g., two different paraffinic hydrocarbons) and forming a mixture thereof, a temperature stabilizing range can be adjusted over a wide range for any particular application. According to some embodiments of the present invention, the mixture of two or more different substances may exhibit two or more distinct transition temperatures or a single modified transition temperature.

[0076] According to an embodiment of the present invention, the microcapsules comprise a shell and a core. According to another embodiment, the core comprises phase-change material. The shell provides at least one of encapsulating, containing, surrounding and absorbing the phase-change material. This shell may facilitate handling of the phase-change material while offering a degree of protection to the phase-change material during manufacture of the fibers (e.g., protection from high temperatures or shear forces). Various materials are suitable for the microcapsules shells, including synthetic polymers, such as formaldehyde-based ones, isocyanate, amines, carboxylic acid derivatives, natural materials such as gelatin or cellulose and acrylic polymers, and others such as silica. The shell polymers can be thermoplastic or thermoset, crosslinked or uncrosslinked, soft or hard, flexible or rigid. According to an embodiment of the present invention, the shell is formed from formaldehyde-based polymers, silica particles, or acrylic polymers precursors such as acrylic acid, methacrylic acid, formaldehyde and a silica precursor, such as described in patents EP1321182 and US6716526 and patent applications WO2004092299 and WO2005105291, the details of which are incorporated by reference in their entirety into the present disclosure.

[0077] The stable suspension of the present invention comprises a solvent capable of dissolving a polymer to be included in a temperature-regulating fabric, or capable of dissolving a precursor of such polymer. Any polymer is suitable for the present invention, if it is incorporated in such temperature-regulating fabric. According to another embodiment of the present invention, the polymer is at least one of acrylonitrile-based polymers and cellulose-based polymer. Any solvent

capable of dissolving the polymer to be incorporated or its precursor is suitable. According to an embodiment of the invention the solvent is an aqueous solution. According to another embodiment of the invention the aqueous solution comprises at least one solute, such as sodium base, sodium thiocyanate, zinc chloride, acetone, DMF, NMP and nitric acid. According to another embodiment of the invention the solute concentration in the solvent on microcapsules-free basis is in a range of between about 0.5 percent and about 90 percent by weight. According to another embodiment of the invention the solute concentration in the solvent on microcapsules-free basis is in a range of between about 1 percent and about 70 percent by weight.

[0078] The stable suspension of the present invention comprises said solvent and said phase-change material-comprising microcapsules. According to an embodiment of the invention the microcapsules form between about 5 and about 50 percent by weight of the suspension. According to an embodiment of the invention the microcapsules form between about 10 and about 30 percent by weight of the suspension.

[0079] A characteristic of the stable suspension of the present invention, comprising said solvent and said microcapsules is that it is stable for at least 20 hours. According to an embodiment of the invention, the stable suspension is stable for at least 40 hours. As used herein the term “stable suspension” can refer to a suspension wherein the majority of the microcapsules are evenly dispersed. As used herein the term “evenly dispersed” refers to a state where the majority of the microcapsules are not agglomerated. The term “stable suspension” can also refer to that suspension, wherein the majority of the microcapsules are not agglomerated. The term can also refer to that suspension, wherein the majority of the microcapsules do not float on top of the solvent nor sink to its bottom. The term can also refer to that suspension, wherein no creaming is observed.

[0080] According to yet another embodiment of the invention, the stable suspension comprises an additive selected from a group consisting of defoaming agents, wetting agents, flow-control agents, dispersing agents and surfactants. Suitable additives include:

- a) Flow control and rheological agents, such as water soluble polymers, water insoluble polymers, clays, microcrystalline cellulose aerosols;

- b) Dispersing agents, such as anionic, cationic, amphoteric and nonionic surfactants, derivatives of polyacrylic acids , low or high molecular weight unsaturated acidic polycarboxylic acid polyester , polyquaternary ammonium compounds, polycarboxylic acids , salts of long chain polyamine amides, and alkylolammonium salt of a block copolymer with acidic groups;
- c) Wetting agents, such as polyether modified poly-dimethyl-siloxane (such as BYK-348, BYK-346 ,BYK-333 from Byk Chemie) .

[0081] In general, the wetting agents may be selected from a group consisting of silicon compounds, fluorine compounds, polyglycols, fatty acids, fatty amides, fatty alcohols and their esters and ethers. Suitable agents can also comprises various mixtures, blends and copolymers from the above list.

[0082] In preferred embodiments of the present invention said first suspension further comprises at least one compound with some functional aspect, said functionality may include, for example, fire retardation, bioactivity, antimicrobial activity, odor resistance, UV absorption, moisture management and resistance to water, grease, dirt and/or stain. According to an embodiment of the invention, functional compounds and functionality are selected from a group consisting of those set forth in Table 3 as well as any similar or related functions.

	<p>shell, or acid groups on the textile substrate.</p> <p>Chitosan is an effective natural antimicrobial agent derived from Chitin, a major component in crustacean shells.</p> <p>Castor oil derivatives based on Undecylene acid or Undecynol. Products such as Undecylenoxy polyethylene glycol acrylate or methacrylate.</p>
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[0083] According to one embodiment of the invention, the functional compound is dissolved in the stable suspension. According to another embodiment, the functional compound is encapsulated in shells. According to a related embodiment, the shell is similar in properties to the shells of the phase-change material microcapsules. According to another embodiment, the shell of the functional-compound microcapsules is made of the same material as the shell of phase-change material microcapsules. According to a related embodiment, the functional compound is encapsulated and the microcapsules are evenly dispersed in the stable suspension of the invention.

[0084] According to an embodiment of the present invention, the stable suspension is kept at a controlled temperature. According to a related embodiment, that temperature is in the range between about 15°C and about 120°C. According to another embodiment, the temperature is kept at a temperature between about 20°C and about 50°C. According to another embodiment, the stable suspension is kept under low-shear mixing. According to an embodiment of the solution, low-shear mixing is mixing or agitation with paddles or propellers at shear rates of 1-100 sec⁻¹, preferably 1-10 sec⁻¹.

[0085] According the present invention, the stable suspension, which comprises the solvent and the plurality of the microcapsules, is used for the production of temperature-regulating, polymer comprising fabric.

[0086] According to another embodiment of the present invention, that polymer is cellulose-based, the solvent of the stable suspension comprises water and sodium hydroxide, the concentration of the sodium hydroxide ranges between about one percent and about 8 percent by weight of the solvent and the microcapsules form between about 15 percent and about 50 percent by weight of said suspension. According to a related embodiment, the polymer is cellulose-

based, the solvent of the stable suspension comprises water and sodium hydroxide, the concentration of the sodium hydroxide ranges between about 1.5 percent and about 3 percent by weight of the solvent and the microcapsules form between about 15 percent and about 30 percent by weight of said suspension and the suspension is kept at an ambient temperature.

[0087] Aspects of the present invention also provides a fabric produced by a method using the stable suspension. More specifically, the provided fabric is a commercial product. According to an embodiment of the invention the fabric or commercial product comprises a fiber or plurality of fibers.

[0088] According to another embodiment of the invention the stable suspension comprises at least one additive selected from a group consisting of defoaming agents, flow-control agents, wetting and dispersing agents and surfactants, e.g. the ones listed above. Optionally, a suitable additive is present in the third suspension, when used to provide the microcapsules. For example, the additive is used in the process of producing the microcapsules and remain in a suspension generated during that production, which suspension is used as the third suspension, as such or after some modification. According to another embodiment, the additive is added as such or in any combination, e.g. solution, to the provided components, e.g. the third suspension or the solvent prior to mixing, and/or added after mixing.

[0089] According to yet another embodiment of the invention, during mixing and/or after it, the stable (first) suspension is kept at a temperature in the range between about ambient and about 120°C. According to another embodiment, during mixing and/or after it, the stable suspension is kept under low-shear mixing.

[0090] According to an embodiment of the invention there is also provided a method of manufacturing polymer-containing temperature-regulating fabric, said method comprising providing and incorporating at least one functional compound with functionality selected from a group consisting of fire retardation, bioactivity, antimicrobial, odor resistance and UV absorption as described hereinafter. According to an embodiment of the invention, functional compounds and functionality are selected from the group consisting of those set forth in Table 3. According to one embodiment of the invention, the functional compound is provided in a dissolved form.

According to another embodiment, the functional compound is encapsulated in shells. According to a related embodiment, the shell is similar in properties to the shells of the phase-change material microcapsules. According to another embodiment, the shell of the functional-compound microcapsules is made of the same material as the shell of phase-change material microcapsules. Any form of provided the functional material and/or its microcapsules is suitable, e.g. in the stable first solution or with the polymer and/or precursor prior to combining those or after such combining. Incorporating the functional compound and/or its microcapsules may use any known method, e.g. mixing. According to an embodiment of the invention, the functional compound is encapsulated and its microcapsules are evenly dispersed in the second suspension. According to an embodiment of the invention, the formed second suspension comprises microcapsules of both phase-change material and functional compounds and both microcapsules are evenly dispersed in the second suspension. According to another embodiment the functional material is provided in the phase-change material-comprising microcapsules.

[0091] According to another embodiment of the invention the method for manufacturing of polymer-containing temperature-regulating fabric comprises the step of converting the second suspension, as such, or with additional components to said fabric. For example, the second suspension is coated by known methods on surfaces, where they is further treated, e.g. evaporation of solvent, precipitation and/or polymerization of components included therein. Also possible is converting the second suspension into polymer pellets, wherein the microcapsules are evenly dispersed, which pellets is then converted to commercial products. The method for manufacturing of polymer-containing temperature-regulating fabric may also comprise the step of converting the second suspension into fiber. According to an embodiment of the invention, the microcapsule-containing, phase-change material is evenly dispersed in said manufactured fiber.

[0092] Many fabrics are made from synthetic fibers. Conventionally, two processes are used to manufacture synthetic fibers: a solution spinning process and a melt spinning process. The solution spinning process is generally used to form acrylic or regenerated cellulosic fibers, while the melt spinning process is generally used to form nylon fibers, polyester fibers, polypropylene fibers, and other similar type fibers. According to an embodiment of the invention the microcapsules-comprising fiber is produced by the melt spinning process, wherein a molten polymer and microcapsules are provided, spun and cooled for solidification. Polymer pellets,

wherein the microcapsules are evenly dispersed, are useful for such fiber production via melt spinning. According to a related embodiment, the shell of the microcapsules used for such melt spinning is made out of silica.

[0093] According to an embodiment of the invention the fiber is manufactured by means of solution spinning involving spinning a feed solution comprising polymer and/or polymer precursor, phase-change material-microcapsules and optionally also at least one agent, surfactant or functional compound, as described above. According to another embodiment of the invention the feed solution may comprises the above-described second solution. According to an embodiment of the invention, a stable suspension is manufactured, as described above, and stored in a suitable first vessel at the above-specified temperature range and under the above-specified mixing. The amount of produced stable suspension is suitable for at least 20 hours of fiber production, preferably for at least 40 hours. A solution of the polymer and/or precursor is also produced and kept in a second vessel. A feed solution is generated by mixing a suspension from said first vessel with solution from said second vessel, which is then spun through at least one spinneret and further treated, e.g. via, optionally polymerization, dry spinning or wet spinning. In the wet spinning process the spinnerets are submerged in or held very close to a chemical bath and as the filaments emerge, they contact the chemical bath, and they precipitate from solution and solidify. In the dry spinning process instead of precipitating the polymer by dilution or chemical reaction, solidification is achieved by evaporating the solvent in a stream of air or inert gas. For more details, see *Acrylic Fibers* by R. Cox in *Synthetic Fibers: Nylon, Polyester, Acrylic, Polyolefins*, Woodhead Publishing ISBN 1 85573 488 1 and U.S patents; 5686034, 6258304, 6333108 and 6538130, G.B patent 2412083, and WO0231236, the relevant teaching of which is incorporated here by reference.

[0094] According to an embodiment of the invention the fiber is acrylonitrile-based. According to said embodiment, the solution in the second vessel is preferably comprised of between about 5 and about 20 percent by weight solute in a solvent. In especially preferred embodiments of the present invention the solution in the vessel comprises between about 10 and about 15 percent by weight solute in solvent. The solute, on a dry basis, is preferably made from between about 80 and about 100 percent by weight of acrylonitrile monomer; between about 0 and about 20 percent by weight of neutral monomer, e.g. at least one of methyl acrylate, vinyl acetate, methyl

methacrylate and acrylamide, and between about 0 and about 2 percent by weight of acid comonomer, e.g. sodium styrene sulphonate, sodium methallyl sulphonate, sodium 2-methyl-2acrylamidopropane sulphonate and itaconic acid.

[0095] In especially preferred embodiments of the present invention the solute, on a dry basis, is made from between about 90 and about 95 percent by weight of acrylonitrile monomer and between about 0 and about 14 percent by weight of said neutral monomer.

[0096] According to another embodiment the fiber is a modacrylic fiber, the proportion of acrylonitrile in the solute is smaller and the solute contains also another comonomer, typically halogenated ethylenically unsaturated molecules. The solvent of the solution in the second vessel, according to an embodiment of the invention, is an aqueous solution of sodium thiocyanate, e.g. with sodium thiocyanate concentration in a range between about 40 and about 60 percent by weight. According to another embodiment of the invention, e.g. in the case of modacrylic fiber, the solvent is a solution of acetone. That solution is prepared by known methods, such as dissolving the polymer by adding it slowly, with stirring, to cold solvent and then raising the temperature to complete dissolution. The solution in the second vessel is kept at a temperature of about ambient.

[0097] According to an embodiment of the invention the stable suspension in the first vessel comprises an aqueous solution of sodium thiocyanate as a solvent. The concentration of the sodium thiocyanate in that solution is between about 40 and about 60 percent by weight. The stable suspension comprises microcapsules with a shell and a core, the shell forming between about 5 and about 40 percent by weight of the microcapsules. The shell is made from a compound such as, silica and formaldehyde polymer. The core comprises a phase-change material, e.g. a straight-chain or a branched-chain hydrocarbon with 15 to 25 carbon atoms. Typically, the microcapsules are of a size (largest dimension) of up to about 2 microns. The microcapsules form between about 5 and about 30 percent by weight of the suspension, which optionally also contains a viscosity modifier, e.g. as listed above. The stable suspension is prepared by gradually adding solvent to microcapsules suspension of about 50% in aqueous medium, while gently mixed. The stable suspension is kept in the first vessel at a temperature of about ambient under gentle low-shear mixing.

[0098] According to yet another embodiment of the invention, a feed solution is produced by mixing a solution from the second vessel with a suspension from the first vessel (optionally filtered) at a relative rate suitable to yield a concentration of 2-50% mPCM on polymer dry weight basis, more preferably 5-20%. The feed solution is then spun by dry spinning or wet spinning (e.g. wherein the spinneret is submerged in or held very close to a relatively dilute aqueous solution) to form acrylic fibers containing microcapsules with phase-change material. According to an embodiment of the invention the fiber contains between about 5 and about 20 percent by weight of evenly dispersed microcapsules. Those fibers are then used for the production of various temperature-adaptable commercial products, such as woven fabrics, knit fabrics, and nonwoven fabrics, e.g. ones with enthalpy between about 1 J/g and about 50 J/g.

[0099] According to another embodiment of the invention the fiber is cellulose-based. According to that embodiment, the solution in the second vessel is composed of between about 5 and about 15 percent by weight solute in a solvent, preferably between about 8 and about 11 percent by weight. The composition of the solute, on a dry basis, is between about 5 and about 15 percent of the aqueous sodium hydroxide solution solvent. The solvent of the solution in the second vessel, according to an embodiment of the invention, is an aqueous solution of sodium hydroxide, e.g. with sodium hydroxide concentration in a range between about 4 and about 10 percent by weight. That solution is prepared by known methods, such as dissolving the sodium hydroxide in water and adding cellulose. Preferably the dry cellulose is added with stirring, to prevent cellulose powder from sticking and forming clumps, to the correct amount of water and dissolved sodium hydroxide. This is then stirred until the cellulose is dissolved and the solution is clear. The solution in the second vessel is kept at a temperature of about ambient

[00100] According to an embodiment of the invention the stable suspension in the first vessel comprises an aqueous solution of sodium hydroxide as a solvent. The concentration of the sodium hydroxide in that solution is between about 1 and about 5 percent by weight. According to an embodiment of the invention the pH of the solvent is at least 10, preferably, at least 11.5. The stable suspension comprises microcapsules with a shell and a core, the shell forming between about 5 and about 40 percent by weight of the microcapsules. The shell is made from at least one compound such as, silica, acrylic acid, its derivative, methacrylic acid and its

derivatives. The core comprises a phase-change material, e.g. a straight-chain or a branched-chain hydrocarbon with 15 to 25 carbon atoms. Typically, the microcapsules are of a size (largest dimension) of up to about 2 microns. The microcapsules form between about 10 and about 50 percent by weight of the suspension, which optionally also contains a viscosity modifier, e.g. cellulose, cellulose derivatives, acid functional polymers, polyglycols, polysaccharide and polyvinyl alcohol. The stable suspension is prepared by gradually adding the solvent to microcapsules suspension of about 50% in an aqueous medium, while gently mixed. The stable suspension is kept in the first vessel at an ambient temperature, under gentle low-shear mixing.

[00101] According to an embodiment of the invention, a feed solution is produced by mixing a solution from the second vessel with a suspension from the first sample (optionally filtered) at a relative rate suitable to yield the desired % of mPCM on cellulose. The feed solution is then spun by dry spinning or wet spinning (e.g. by spinnerets submerged in or held very close to a solution of an acid, e.g. sulfuric acid) to form rayon or viscose fiber comprising microcapsules with phase-change material. According to an embodiment of the invention the fiber contains between about 5 and about 40 percent by weight of evenly dispersed microcapsules. Those fibers is then used for the production of various temperature-adaptable commercial products -, such as woven fabrics, knit fabrics, and nonwoven fabrics, e.g. ones with enthalpy between about 1 J/g and about 50 J/g.

[00102] While the invention will now be described in connection with certain preferred embodiments in the following examples and with reference to the accompanying figures so that aspects thereof may be more fully understood and appreciated, it is not intended to limit the invention to these particular embodiments. On the contrary, it is intended to cover all alternatives, modifications and equivalents as may be included within the scope of the invention as defined by the appended claims. Thus, the following examples which include preferred embodiments will serve to illustrate the practice of this invention, it being understood that the particulars shown are by way of example and for purposes of illustrative discussion of preferred embodiments of the present invention only and are presented in the cause of providing what is believed to be the most useful and readily understood description of formulation procedures as well as of the principles and conceptual aspects of the invention.

[00103] Example 1 - stable suspension of mPCM for acrylic fibers

To 100.0 kilograms of formaldehyde-based shell microcapsules (microencapsulated n-hydrocarbons, 115 J/g latent heat, 50 percent microcapsules, available from Ciba Specialty Chemical Co., Bradford, United Kingdom) is added with stirring 121.5 kilograms of water then 178.5 kilograms sodium thiocyanate. This yields a stable suspension containing 12.5% microcapsules with a sodium thiocyanate:water ratio of 51/49. This suspension of microcapsules is stable against agglomeration >21 hours.

[00104] Example 2 - stable suspension of mPCM for acrylic fibers

To 100.0 kilograms of formaldehyde-based shell microcapsules (microencapsulated n-hydrocarbons, 115 J/g latent heat, 50 percent microcapsules, available from Ciba Specialty Chemical Co., Bradford, United Kingdom) is added with stirring a premixed solution of 121.5 kilograms of water and 178.5 kilograms sodium thiocyanate. This yields a stable suspension containing 12.5% microcapsules with a sodium thiocyanate:water ratio of 51/49. This suspension of microcapsules is stable against agglomeration >21 hours.

[00105] Example 3 - stable suspension of mPCM for Rayon , Viscose or Cellulose fibers

To 100.0 kilograms of polyacrylic shell microcapsules (microencapsulated octadecane, 175 J/g latent heat, 45 percent microcapsules, available from Ciba Specialty Chemical Co., Bradford, United Kingdom) is added with stirring 100.0 kilograms of water, then 5.2 kilograms of a 50% NaOH/Water solution. This yields a stable suspension containing 21.95% microcapsules at a pH of 12.8. This suspension of microcapsules is stable against agglomeration >24 hours as shown by original slurry particle size distribution (Fig. 1) and after 24 hours (Fig. 2). As will be noted, there is no significant difference between the distribution when the suspension is first formed and the distribution after 24 hours. This stable suspension provided for excellent spinning performance, no line stoppage over multiple days, no filter plugging or blockage and was used to spin 1.7dtex viscose fiber.

[00106] Example 4 - Unstable suspension of mPCM for Rayon, Viscose or Cellulose fibers

To 100.0 kilograms of polyacrylic shell microcapsules (microencapsulated octadecane, 175 J/g latent heat, 45 percent microcapsules, available from Ciba Specialty Chemical Co., Bradford, United Kingdom) is added with stirring 81.8 kilograms of water, then 1.8 kilograms of a 50% NaOH/Water solution. This yields an unstable suspension containing 25.0% microcapsules at a pH of 9.5. This unstable suspension caused pressure buildup, filter blockage and line shutdown after 20 min.

[00107] Example 5 - stable suspension of mPCM for Lyocell fibers

0.90 g of deionized water and 0.20 g of water-wetted microcapsules containing a phase-change material (microencapsulated paraffin PCM, 120 J/g latent heat, 50 percent microcapsules, available from Ciba Specialty Chemical Co., Bradford, United Kingdom) were combined in a 20 ml glass vial. Next, 8.00 g of N-methyl morpholine oxide solvent (97 percent NMMO, available from Aldrich Chemical Co., Milwaukee, Wis.) were added to yield a solution with 1.1 percent by weight of mPCM solids. The vial was placed in a 125°C oven and periodically mixed until its contents were homogenously mixed and the solvent is melted. This solution can be used immediately or it can be cooled/solidified for storage then reheated. This cycle can be repeated numerous times.

[00108] Example 6 - stable suspension of mPCM for Lyocell fibers

0.90 g of deionized water and 0.20 g of water-wetted microcapsules containing a phase-change material (microencapsulated paraffin PCM, 120 J/g latent heat, 50 percent microcapsules, available from Ciba Specialty Chemical Co., Bradford, United Kingdom) were combined in a 20 ml glass vial. Next, 8.00 g of N-methyl morpholine oxide solvent (97 percent NMMO, available from Aldrich Chemical Co., Milwaukee, Wis.) and 0.90 g of microcrystalline cellulose (available from Aldrich Chemical Co., Milwaukee, Wis.) were added to yield a solution with 10 percent by weight of solids. The solids included a 90/10 weight ratio of cellulose/microcapsules containing the phase-change material. The vial was placed in a 125°C oven and periodically mixed until its contents were homogenously mixed and melted. This solution can be used immediately or it can be cooled/solidified for storage then reheated. This cycle can be repeated numerous times.

[00109] It will be evident to those skilled in the art that the invention is not limited to the details of the foregoing **illustrative** examples and that the present invention may be embodied in other specific forms without departing from the essential attributes thereof, and it is therefore desired that the present embodiments and examples be considered in all respects as illustrative and not restrictive, reference being made to the appended claims, rather than to the foregoing description, and all changes which come within the meaning and range of equivalency of the claims are therefore intended to be embraced therein.

Claims

1. A stable first suspension for the production of a temperature-regulating, polymer-containing fabric, said suspension comprising a solvent and a plurality of microcapsules containing at least one phase-change material, wherein said microcapsules are to be incorporated in said polymer-containing fabric, and wherein said stable first suspension is characterized in that:
 - (i) said solvent is capable of dissolving a fabric-forming component selected from the group consisting of at least one of said polymer and precursors thereof; and
 - (ii) said suspension is stable for at least about 20 hours.
2. The first suspension of Claim 1, further characterized in that at least about 95% of said microcapsules stay intact in said suspension for at least about 20 hours.
3. The first suspension of Claim 1, wherein said fabric is formed from at least one type of polymeric fiber.
4. The first suspension of Claim 1, wherein said polymer is selected from the group consisting of acrylonitrile-based polymers, cellulose-based polymers, polyester-based polymers, polyamide-based polymers and polyolefin based polymers.
5. The first suspension of Claim 1, wherein said solvent is an aqueous solution.
6. The first suspension of Claim 1, further comprising at least one further component selected from the group consisting of a defoaming agent, a flow-control agent, a wetting agent, a dispersing agent and surfactant.
7. The first suspension of Claim 5, wherein said aqueous solutions comprise at least one further component selected from the group consisting of sodium bases, sodium thiocyanate, zinc chloride, n-methyl morpholine oxide, ammonia, copper sulfate, nitric acid, acetone, DMF and NMP.

8. The first suspension of Claim 1, wherein said microcapsules comprise a shell and a core and wherein said shell is formed from at least one component selected from the group consisting of acrylic acid and derivatives thereof, methacrylic acid and derivatives thereof, formaldehyde, isocyanate, urea, carboxylic acid derivatives, silica precursor and gelatin.
9. The first suspension of Claim 1, wherein said microcapsules comprise a shell and a core and wherein said core comprises a phase-change material selected from the group consisting of octadecane, straight-chain hydrocarbons with 15 to 25 carbon atoms, and branched-chain hydrocarbons with 15 to 25 carbon atoms.
10. The first suspension of Claim 1, wherein said microcapsules comprise a shell and a core and the weight ratio between said shell and core is in the range between about 0.5:9.5 and about 4:6.
11. The first suspension of Claim 1, wherein said microcapsules form between about 5 and about 40 percent by weight of said suspension.
12. The first suspension of Claim 1, wherein said microcapsules have a maximum linear dimension ranging between about 0.1 and about 20 micron.
13. The first suspension of Claim 1, further comprising at least one compound with functionality, said functionality being selected from the group consisting of fire retardation, bioactivity, antimicrobial activity, odor resistance, UV absorption, moisture management and resistance to water, grease, dirt and/or stain.
14. The first suspension of Claim 1, when kept at a temperature in the range between about ambient and 120 °C.
15. The first suspension of Claim 1, wherein said polymer is acrylonitrile-based, wherein said solvent comprises water and sodium thiocyanate, wherein the concentration of the sodium thiocyanate ranges between about 40 and about 60 percent by weight of the

- solvent and wherein said microcapsules form between about 5 and about 30 percent by weight of said suspension.
16. The first suspension of Claim 1, wherein said polymer is cellulose-based, wherein said solvent comprises water and sodium hydroxide, wherein the concentration of the sodium hydroxide ranges between about 1 percent and about 5 percent and wherein said microcapsules form between about 5 and about 30 percent of said suspension.
 17. A second suspension formed from the first suspension of Claim 1, in combination with at least one fabric-forming component selected from the group consisting of a polymer and a precursor thereof.
 18. The second suspension of Claim 17, wherein said microcapsules are evenly dispersed throughout said suspension.
 19. The second suspension of Claim 17, wherein the said second suspension is stable for at least about 20 hours.
 20. A commercial product comprising said fabric of Claim 1.
 21. A method for manufacturing of said first suspension of Claim 1, comprising the steps of:
 - (i) providing microcapsules containing a phase-change material;
 - (ii) providing a solvent capable of dissolving a fabric-forming component selected from the group consisting of at least one of said polymer and precursors thereof; and
 - (iii) mixing said solvent and said microcapsules to form said first suspension.
 22. The method of Claim 21, wherein said microcapsules are provided in a vessel and said solvent is added to said microcapsules in said vessel to form said first suspension.
 23. The method of Claim 21, wherein said solvent is an aqueous solution.

24. The method of Claim 21 further comprising the step of adding at least one further component selected from the group consisting of a defoaming agent, a flow-control agent, a wetting agent, a dispersing agent and a surfactant.
25. The method of Claim 21, wherein said microcapsules are provided as a third suspension of microcapsules in a fluid.
26. The method of Claim 25, wherein said third suspension of microcapsules in a fluid is provided in a vessel and said solvent is added to said microcapsules in said vessel to form said first suspension.
27. The method of Claim 25, wherein said fluid is an aqueous medium.
28. The method of Claim 25, wherein the viscosity of said third suspension is in the range of between about 100 and about 3000 cps.
29. The method of Claim 25, wherein said third suspension is provided at a temperature in the range of between about ambient and 120 °C.
30. The method of Claim 21, wherein said solvent is provided at a temperature in the range of between about ambient and 120 °C.
31. The method of Claim 21, wherein said first suspension is kept at a temperature in the range of between about ambient and 120 °C.
32. The method of Claim 21, wherein said first suspension is kept under low-shear mixing.
33. A method for the manufacturing of the second suspension of claim 17 comprising the steps of providing at least one fabric-forming component selected from the group

consisting of a polymer and a precursor thereof and combining said component with said first suspension to form said second suspension.

34. The method of claim 33, further comprising wet spinning or dry spinning of said second suspension.
35. The method of claim 34, wherein said suspension is spun to form fibers.
36. The method of claim 35, wherein said fibers are combined with other fibers to form a fabric.
37. Fibers manufactured according to the method of Claim 35.
38. The fibers of Claim 35, wherein said microcapsules are evenly dispersed throughout said fiber.
39. A commercial product comprising said fibers of Claim 35.

1/1

Fig.1

Slurry Particle Size Distribution - Original

PCM

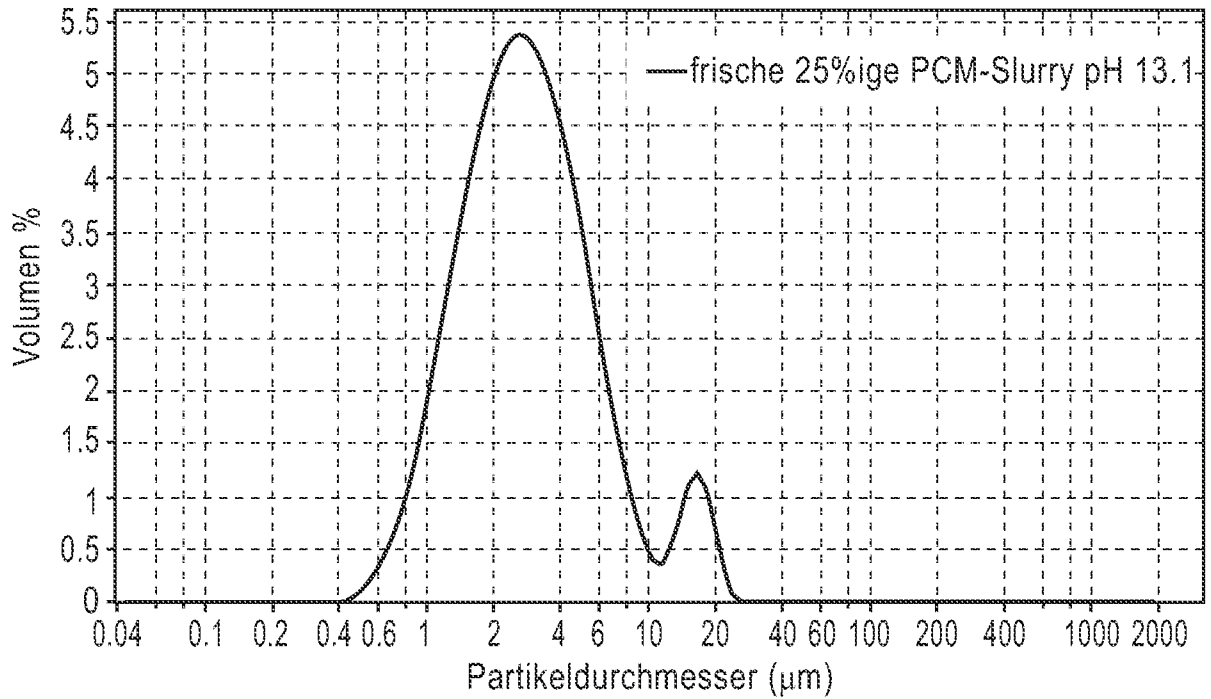
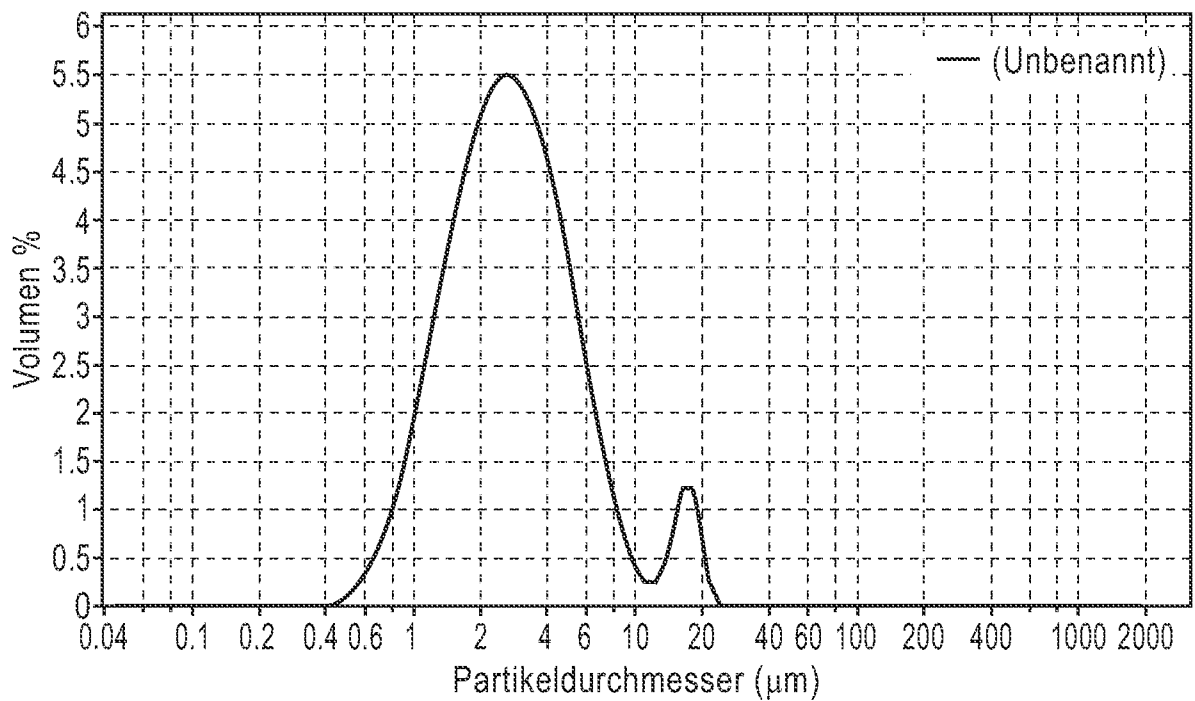


Fig.2


Slurry Particle Size Distribution - 24 hours



INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 07/71373

A. CLASSIFICATION OF SUBJECT MATTER IPC(8) - D06M 23/12, D06M 23/10, D04H 1/00 (2007.10) USPC - 8/115.51 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) USPC - 8/115.51 Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched USPC - 8/115.51, 28/299, 8/\$, 28/\$ Search Terms Below Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) PubWEST (USPT, PGPB, EPAB, JPAB); google.com Search Terms Used: microcapsules, phase-change, fabric, suspension, stable		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X -- Y	US 6,660,667 B2 (Zuckerman et al.) 04 July 2006 (04.07.2006) entire document, especially Abstract; col. 1, ln. 17-22; col. 2, ln. 42-48; col. 4, ln. 20-24; col. 4, ln. 34-51; col. 5, ln. 5-40; col. 5, ln. 48-48-56; col. 6, ln. 10-21; col. 6, ln. 39-57; col. 7, Table I; col. 7, ln. 48-60; col. 8, Table II; col. 9, ln. 1-4; col. 9, ln. 10-12; col. 9, Table IV; col. 10, ln. 17-26; col. 12, Table VII; col. 13, ln. 7-22; col. 6, ln. 10-20	1-6, 8, 9, 11-14, 17-39 7, 10, 15, 16
Y	US 5,596,051 A (Jahns et al.) 21 January 1997 (21.01.1997) entire document, especially Abstract; col. 7, ln. 10-30	7, 10, 16
Y	US 4,367,191 A (Cuculo et al.) 04 January 1983 (04.01.1983) entire document, especially Abstract; col. 2, ln. 56-57	15
A	US 4,464,271 A (Munteanu et al.) 07 August 1984 (07.08.1984) entire document, especially Abstract; col. 18, ln. 58 to col. 19, ln. 7	17, 26
<input type="checkbox"/> Further documents are listed in the continuation of Box C. <input type="checkbox"/>		
* Special categories of cited documents: "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "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 "&" document member of the same patent family		
Date of the actual completion of the international search 14 December 2007 (14.12.2007)		Date of mailing of the international search report 10 JAN 2008
Name and mailing address of the ISA/US Mail Stop PCT, Attn: ISA/US, Commissioner for Patents P.O. Box 1450, Alexandria, Virginia 22313-1450 Facsimile No. 571-273-3201		Authorized officer: Lee W. Young  PCT Helpdesk: 571-272-4300 PCT OSP: 571-272-7774