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PROCESS OF PRODUCING VISCOSE RAYON

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This invention relates to the production of shaped bodies of regenerated cellulose from viscose and more particularly to filaments and fibers of regenerated cellulose from viscose.

The present application is a continuation in part of application Serial No. 466,676, filed November 3, 1954.

In the conventional methods of producing shaped bodies of regenerated cellulose from viscose, a suitable cellulosic material such as purified cotton linters, wood pulp, mixtures thereof, and the like is first converted to an alkali cellulose by treatment with a caustic soda solution and after shredding the treated cellulose material, it is allowed to age. The aged alkali cellulose is then converted to a xanthate by treatment with carbon disulfide. The cellulose xanthate is subsequently dissolved in a caustic soda solution in an amount calculated to provide a viscose of the desired cellulose and alkali content. After filtration, the viscose solution is allowed to ripen and is subsequently extruded through a shaped orifice into a suitable coagulating and regenerating bath.

In the production of shaped bodies such as filaments, the viscose solution is extruded through a spinneret into a coagulating and regenerating bath consisting of an aqueous acid solution containing zinc sulfate. The filament may subsequently be passed through a hot aqueous bath where it is stretched to improve its properties such as tensile strength. The filament may then be passed through a dilute aqueous solution of sulfuric acid and sodium sulfate to complete the regeneration of the cellulose, in case it is not completely regenerated upon leaving the stretching stage. The filament is subsequently subjected to washing purification, bleaching, possibly other treating operations and drying, being collected either before or after these treatments.

The filaments as formed by the conventional methods, consist of a skin or outer shell portion and a core portion with a sharp line of demarkation between the two. The cross section of the filaments exhibits a very irregular or crenulated exterior surface when even small amounts of zinc salts or certain other polyvalent metal salts are present in the spinning bath. The skin and core portions of the filament represent differences in structure and these different portions possess different swelling and staining characteristics, the latter permitting a ready identification of skin and core. The sharply irregular and crenulated surface structure has a relatively low abrasion resistance and readily picks up foreign particles such as dirt. Although the core portion possesses a relatively high tensile strength, it has a low abrasion resistance and a low flex-life, is subject to fibrillation and is relatively stiff.

It has now been discovered that the presence of small amounts of alkali-soluble alkylene oxide adducts of castor oil, hydrogenated castor oil and mixtures of castor oil and hydrogenated castor oil in viscose results in the production of shaped bodies of regenerated cellulose such as filaments, films, sheets and the like composed of all skin and having improved properties and characteristics providing that the amount of the adduct is maintained with certain limits and the composition of the spinning bath is maintained within certain composition limits which will be defined hereinafter. The most readily distinguishable characteristics as compared to conventional filaments include a smooth, non-crenulated surface and the filaments consist substantially entirely of skin.

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This invention contemplates the use of such compounds as are more technically classed as polyoxyalkylene glycol ethers of castor oil and of hydrogenated castor oil; for example, the ethers of ethylene, propylene and butylene glycols. It is obvious that for all practical purposes considering cost, ease of preparation, commercial availability and solubility in water and alkali solutions such as a 6 percent caustic solution, the polyoxyethylene glycol ethers or ethylene oxide adducts of castor oil and of hydrogenated castor oil are preferred. Accordingly, the invention will be illustrated by reference to the polyoxyethylene glycol ethers.

The polyoxyethylene content of the adducts or ethers may vary from about 20 to 200 and more ethylene oxide units per molecule of castor oil or of hydrogenated castor oil, the preferred ethers containing from about 30 to about 100 ethylene oxide units per molecule. Theoretically, such an ether containing 30 ehrylene oxide units would consist of an ether wherein the hydroxyl group of each of the ricinoleic acid radicals has been replaced by a chain of 10 ethylene oxide units. It is to be understood that for the purposes of this invention each of the chains of ethylene oxide units need not be identical. The production of all skin products requires that certain minimum amounts of the ether be in solution in the viscose. Therefore, the minimum number of units of an alkylene oxide such as ethylene oxide required in the ether is that number which imparts to the castor oil ether or hydrogenated castor oil ether sufficient alkali solubility whereby the minimum amount of the ether can be dissolved in the viscose. The ether may be conveniently added to the viscose in the form of a solution in alkali or in water. It is preferred to employ ethers having an alkylene oxide content in excess of the minimum requirement whereby the ether may be readily dissolved in and distributed uniformly throughout the viscose during the usual mixing stage employed in the production of the viscose.

The amount of the ether or adduct which is incorporated in the viscose must be at least about 0.2 percent by weight of the cellulose and may vary up to about 4 percent, preferably, the amount varies from about 0.5 percent to 3 percent. Lesser amounts do not result in the production of products consisting entirely of skin and greater amounts affect adversely the physical properties of the products. Amounts within the preferred range are most effective in enhancing the characteristics and properties of the products. The ether or adduct may be added at any desired stage in the production of the viscose, preferably being added after the cellulose xanthate has been dissolved in the caustic solution.

The viscose may contain from about 4 percent to about 10 percent cellulose, the particular source of the cellulose being selected for the ultimate use of the regenerated cellulose product. The caustic soda content may be from about 4 percent to about 11 percent and the carbon disulfide content may be from about 32 percent to about 60 percent based upon the weight of the cellulose. The modified viscose, that is, a viscose containing a small amount of the ether or adduct, should have a salt test number or index above about 6 and preferably about 8.0 or higher at the time of spinning or extrusion. The salt test is an indication of both the amount of carbon disulfide added to the viscose and the degree of aging. The salt test number is the minimum percent concentration of sodium chloride solution at 18° C. required to coagulate 3 drops of viscose. It is determined by dropping the viscose into a beaker containing about 40 cc. of sodium chloride solution at 18° C. while stirring. Stirring is continued for 90 seconds after the last drop of viscose has been added.

In order to obtain the improvements heretofore mentioned and particularly to obtain all-skin, or substantially all-skin yarn with its resulting advantages, it has been found that in addition to the presence of the polyoxyalkylene glycol ethers of castor oil, the amount of carbon disulfide used in the preparation of the viscose and the acid and zinc contents of the acid spinning bath are important. The presence of the modifier of this invention in a viscose of required carbon disulfide content combined with these spinning baths results in the production of yarns of improved properties such as high tenacity, high abrasion resistance, high fatigue resistance and consisting of filaments composed substantially or entirely of skin.

In practicing the present invention the carbon disulfide added in preparing the viscose should be at least 32 percent based on the bone dry cellulose content, and preferably in the order of 35 percent to 50 percent.

Also, generically and in terms of the industrial art, the spinning bath used in the practice of the present invention is a low acid-high zinc spinning bath. The bath should contain from about 10 percent to about 25 percent sodium sulfate and from about 4 percent to about 15 percent zinc sulfate, preferably from 15 percent to 22 percent sodium sulfate and from 5 to 12 percent zinc sulfate. Other metal sulfates such as iron, manganese, nickel and the like may be present and may replace some of the zinc sulfate. The temperature of the spinning bath may vary from about 25° C. to about 80° C., though at the lower temperatures the higher concentrations of sodium sulfate cannot be used because of the difficulty of solubility. However, at the preferred temperatures of between about 40° C. to about 70° C. solubility is no problem. In the production of the all-skin type filaments, the temperature of the spinning bath is not critical. However, as is well known in the conventional practice in the art, certain of the physical properties such as tensile strength are affected by the temperature of the spinning bath. Thus, in the production of filaments for tire cord purposes in accordance with the method of this invention, the spinning bath is preferably maintained at a temperature between about 40° C. and 65° C. so as to obtain the desired high tensile strength.

The acid content of the spinning bath is balanced against the composition of the viscose. The lower limit of the acid concentration, as is well known in the art, is just above the slubbing point, that is, the concentration at which small slubs of uncoagulated viscose appear in the strand as it leaves the spinning bath. Though for any given spinning conditions a substantially higher percentage of skin will be obtained through the use of the additives of the present invention, in order to get an all-skin yarn the carbon disulfide content of the viscose should preferably be at least 35 percent based on the dry cellulose. Also, the acid content of the spinning bath should preferably not exceed 9 percent while the zinc sulfate should preferably be within the range of 5 percent to 12 percent. However, frequently in commercial spinning practice it is desirable to have a somewhat higher acid content in the spinning bath. It has been found that higher acid contents can be used for any given amount of additive and still exceptionally high-skin and all-skin yarns obtained if the carbon disulfide content of the viscose is increased and the zinc sulfate content of the bath maintained at a reasonable high level such as above 8.0 percent. Thus, even though in practicing the present invention for the production of all-skin products, it is generally preferred that the acid concentration of the bath not exceed 9 percent all-skin products will be obtained at higher acid concentrations up to as high as 11 percent if the carbon disulfide content of the viscose is sufficiently increased and the zinc sulfate of the spinning bath is not appreciably below 8.0 percent. Of course, increasing the amount of additive, within the range specified, also permits, to some degree, the use of higher acid concentrations in the spinning bath. As a general rule it can be stated, in practicing the present invention with the additives mentioned, that if difficulty is encountered in obtaining a 100 percent skin product because of the desire to use a spinning bath having an acid content in excess of 9 percent somewhat higher acid contents can be used by using a viscose of higher carbon disulfide content and by increasing the zinc sulfate content of the bath within the limits indicated.

Though various terminology may be used to indicate the point at which the viscose is sufficiently coagulated to permit uniform withdrawal from the spinneret, for purposes of the present application the term "slubbing point" will be used. This lower acid concentration is readily determined by those skilled in the art through observance of the filaments as formed and has no particular effect on the production of high-skin or all-skin products in practicing the present invention. In general, the lower limit of sulfuric acid required in order to give sufficient coagulation for spinning, for example with a viscose containing 7 percent cellulose and 6 percent caustic, is about 6 percent. Another way of stating this is that an acid concentration of about 6 percent is slightly above the "slubbing point." It is apparent that for viscose with higher caustic content a slightly higher acid concentration would be required due to the partial neutralizing effect of the increased caustic.

It is clear from the above that there is a maximum acid concentration for any specific viscose composition of given carbon disulfide content beyond which substantially all-skin products will not be obtained with the present additives. It is also clear that this maximum acid concentration can be somewhat increased through the use of higher concentrations of zinc sulfate in the spinning bath and by increasing the carbon disulfide content of the viscose. For example, in general, the acid concentration of the spinning baths which are satisfactory for the production of all and substantially all-skin products from a 7 percent cellulose, 6 percent caustic, 35 percent carbon disulfide viscose containing the above mentioned ethers of castor oil, lies between about 6 percent and about 9 percent. The acid concentration may be increased as the amount of additive is increased and also as the salt test of the viscose is increased. There is an upper limit, however, for the acid concentration based upon the amount of modifier and the concentration of caustic in the viscose. All-skin products cannot be obtained if the acid content of the bath is increased above the maximum value even though the amount of added modifier is increased to as much as 4 percent while other conditions are maintained constant. For example, a viscose containing about 7 percent cellulose, about 6 percent caustic soda and, based on the weight of cellulose, about 41 percent carbon disulfide and 2 percent of a polyoxyethylene glycol ether of castor oil containing about 40 ethylene oxide units per molecule and having a salt test of 9 to 10 when extruded into spinning baths containing 16 to 20 percent sodium sulfate, 4 to 8 percent zinc sulfate and sulfuric acid of not more than about 8 percent, results in the production of all-skin filaments. Lesser amounts of sulfuric acid may be employed. Greater amounts of acid result in the production of products having skin and core. At acid concentrations in excess of 9 percent the skin would generally comprise less than 85 percent of the filament. By increasing the carbon disulfide content to 45 percent, however, and increasing the zinc sulfate content of the acid spinning bath to about 10 percent the acid concentration of the spinning bath can be increased to as much as 11 percent and similar results can be obtained.

It has been determined that the maximum permissible acid content of the bath is approximately 0.25 times the carbon disulfide of the viscose as based on the dry cellulose and is preferably maintained between about 0.20 and 0.22 times the carbon disulfide content of the viscose. It is also generally preferred that the acid content of the spinning bath not exceed about 1.35 times the caustic soda content of the viscose.

The presence of the adducts in the viscose retards the coagulation and, therefore, the amount of adduct employed must be reduced at high spinning speeds. Thus, for optimum physical characteristics of an all-skin yarn formed from a viscose as above and at a spinning speed of about 50 meters per minute, the adduct is employed in amounts within the lower portion of the range, for example, about 0.5 percent. The determination of the specific maximum and optimum concentration of acid for any specific viscose, spinning bath and spinning speed is a matter of simple experimentation for those skilled in the art. The extruded viscose must, of course, be immersed or maintained in the spinning bath for a period sufficient to effect relatively complete coagulation of the viscose, that is, the coagulation must be sufficient so that the filaments will not adhere to each other as they are brought together and withdrawn from the bath.

In the production of filaments for such purposes as the fabrication of tire cord, the filaments are preferably stretched after removal from the initial coagulating and regenerating bath. From the initial spinning bath, the filaments may be passed through a hot aqueous bath which may consist of hot water or a dilute acid solution and may be stretched from about 70 percent to about 110 percent, preferably between 85 percent and 100 percent. Yarns for other textile purposes may be stretched as low as 20 percent. The precise amount of stretching will be dependent upon the desired tenacity and other properties and the specific type of product being produced. It is to be understood that the invention is not restricted to the production of filaments and yarns but it is also applicable to other shaped bodies such as sheets, films, tubes and the like. The filaments may then be passed through a final regenerating bath which may contain from about 1 percent to about 5 percent sulfuric acid and from about 1 percent to about 5 percent sodium sulfate with or without small amounts of zinc sulfate if regeneration has not previously been completed.

The treatment following the final regenerating bath, or the stretching operation where regeneration has been completed, may consist of a washing step, a desulfurizing step, the application of a finishing or plasticizing material and drying before or after collecting, or may include other desired and conventional steps such as bleaching and the like. The treatment after regeneration will be dictated by the specific type of shaped body and the proposed use thereof.

Regenerated cellulose filaments prepared from viscose containing the small amounts of the alkali-soluble alkylene oxide adducts of castor oil or of hydrogenated castor oil and spun in the spinning baths of limited acid content have a smooth or noncrenulated surface and consist substantially entirely of skin. Because of the uniformity of structure throughout the filament, the swelling and staining characteristics are uniform throughout the cross section of the filament. Filaments produced pursuant to this invention and consisting entirely of skin have a high toughness and a greater flexing life which may be attributed by the uniformity in skin structure through the filament. Although the twisting of conventional filaments, as in the production of tire cord, results in an appreciable loss of tensile strength, there is appreciably less loss in tensile strength in the production of twisted cords from the filaments consisting entirely of skin. Filaments prepared from viscose containing the polyalkylene oxide adducts of castor oil or of hydrogenated castor oil have a high tensile strength as compared to normal regenerated cellulose filaments, have superior abrasion and fatigue resistance characteristics and have a high flex-life. Such filaments are highly satisfactory for the production of cords for the reinforcement of rubber products such as pneumatic tire casings, but the filaments are not restricted to such uses and may be used for other textile applications.

The invention may be illustrated by reference to the preparation of regenerated cellulose filaments from a viscose containing about 7 percent cellulose, about 6 percent caustic soda, and having a total carbon disulfide content of about 41 percent based on the weight of the cellulose. The viscose solutions were prepared by xanthating alkali cellulose by the introduction of 36 percent carbon disulfide, based on the weight of the cellulose, and churning for about 2½ hours. The cellulose xanthate was then dissolved in caustic soda solution. An additonal 5 percent carbon disulfide, based on the weight of the cellulose, was then added to the mixer and the mass mixed for about one hour. The polyoxyethylene glycol ether of castor oil was added to the caustic soda solution and mixed for about ½ hour. The viscose was then allowed to ripen for about 30 hours at 18° C.

Example I

Approximately 2 percent of a polyoxyethylene glycol ether of castor oil, containing about 40 ethylene oxide units per molecule of castor oil, was added to and incorporated in the viscose as described above. The viscose employed in the spinning of filaments had a salt test of 9. The viscose was extruded through a spinneret to form a 200 denier, 120 filament yarn at a rate of about 22 meters per minute. The coagulating and regenerating bath was maintained at a temperature of about 60° C. and contained 7.3 percent sulfuric acid, 8 percent zinc sulfate and 18 percent sodium sulfate. The yarn was passed over a godet from which it was conducted through a hot water bath maintained at about 95° C. During the travel through the hot water bath, the yarn was stretched approximately 82 percent. The yarn was then collected in a spinning box, washed free of acid and salts and dried.

The individual filaments have a smooth, non-crenulated exterior surface and consist entirely of skin, no core being detectable at high magnification (e.g. 1500×). The filaments of a control yarn spun with the same viscose but without the addition of polyoxyethylene glycol ether of castor oil and spun under the same conditions, exhibit a very irregular and serrated surface and are composed of about 70 percent skin and about 30 percent core with a sharp line of demarkation between the skin and core.

Example II

A viscose solution as described hereinbefore was prepared and 1 percent of a polyoxyethylene glycol ether of castor oil, containing about 81 ethylene oxide units per molecule of castor oil, was added to and incorporated in the viscose. The viscose had a salt test of 9 and was spun into a 200 denier, 120 filament yarn by extrusion into a bath containing 7.8 percent sulfuric acid, 8 percent zinc sulfate and 19 percent sodium sulfate. The bath was maintained at a temperature of 60° C. The extrusion rate was about 22 meters per minute. The water bath was maintained at about 95° C. and the filaments were stretched approximately 82 percent while passing through the hot water. The yarn was collected in a spinning box, washed free of acid and salts and dried.

The individual filaments were readily distinguishable from control filaments prepared from viscose containing no modifier in that they have a smooth, non-crenulated surface and consist entirely of skin. Control filaments have a very irregular and serrated surface and consist of about 70 percent skin and 30 percent core with a sharp line of demarkation between the skin and core.

Example III

A modified viscose as described in Example II was spun at a salt test of 10 to form a 1635 denier, 720 filament yarn for the production of a tire cord. The yarn was stretched about 90 percent, washed free of acid and salts on thread-advancing reels, dried and collected on a cone.

The individual filaments have a smooth, non-crenulated surface and consist entirely of skin while control filaments have a very irregular and serrated surface and consist of 65 percent to 70 percent skin and the balance core with a sharp line of demarkation between the skin and core.

The improvements in the physical properties of viscose rayon and other shaped products obtained by the incorporation of the ethers in the viscose is apparent from the table which follows, the data being also representative of the general improvements for other ethers containing different amounts of ethylene oxide:

	Tenacity, Grams per denier		Elongation, Percent		Skin, percent
	Wet	Dry	Wet	Dry	
Example 1	2.8	3.5	30	25	100
Example 2	2.7	3.4	30	23	100
Control	2.1	2.9	27	20	70
Example 3	3.3	4.6	28.7	12.6	100

One of the properties of viscose rayon which has limited its uses is its relatively high cross-sectional swelling when wet with water, this swelling amounting to from about 65 percent to about 80 percent for rayon produced by conventional methods. Rayon filaments produced in accordance with the method of this invention have an appreciably lower cross-sectional swelling characteristic, the swelling amounting to from about 45 percent to about 60 percent.

If desired, small amounts of the modifier may be added to the spinning bath. Since the ethers are water-soluble, some of the modifier will be leached from the filament and will be present in the bath.

The modifier of this invention may be added to any desired viscose such as those normally used in industry, the specific viscose composition set forth above, being merely for illustrative purposes. The adducts or ethers may be added at any desired stage in the production of the viscose and may be present in the cellulosic raw material provided that the amount present will produce a viscose having the proper proportion of the additive at the time of spinning.

The term skin is employed to designate that portion of regenerated cellulose filaments which is permanently stained or dyed by the following procedure: a microtome section of one or more of the filaments mounted in a wax block is taken and mounted on a slide with Meyer's albumin fixative. After dewaxing in xylene, the section is placed in successive baths of 60 percent and 30 percent alcohol for a few moments each, and it is then stained in 2 percent aqueous solution of Victoria Blue BS conc. (General Dyestuffs Corp.) for 1 to 2 hours. At this point, the entire section is blue. By rinsing the section first in distilled water and then in one or more baths composed of 10 percent water and 90 percent dioxane for a period varying from 5 to 30 minutes depending on the particular filament, the dye is entirely removed from the core, leaving it restricted to the skin areas.

While preferred embodiments of the invention have been disclosed, the description is intended to be illustrative and it is to be understood that changes and variations may be made without departing from the spirit and scope of the invention as defined by the appended claims.

I claim:

1. In a method of producing shaped bodies of regenerated cellulose consisting substantially entirely of skin, the steps which include extruding a viscose having a salt index of at least 6 and containing from about 4 percent to 10 percent cellulose, from about 4 percent to 11 percent caustic soda, from about 32 percent to 60 percent carbon disulfide based on the weight of the cellulose and from about 0.2 percent to about 4 percent, based on the weight of the cellulose, of an alkali-soluble polyoxyalkylene glycol ether of a substance selected from the group consisting of castor oil, hydrogenated castor oil and mixtures of castor oil and hydrogenated castor oil into an aqueous spinning bath containing from about 10 percent to 25 percent sodium sulfate, from about 4 percent to 15 percent zinc sulfate and sulfuric acid, the percent sulfuric acid content of the spinning bath exceeding the slubbing point but not exceeding approximately 0.25 times the percent carbon disulfide content of the viscose, based on the weight of the cellulose in the viscose.

2. The steps in the method as defined in claim 1 wherein the shaped bodies consist of filaments and the ether is a polyoxyethylene glycol ether of castor oil containing from about 30 to 100 ethylene oxide units per molecule.

3. In a method of producing shaped bodies of regenerated cellulose consisting substantially entirely of skin, the steps which include extruding a viscose having a salt index of at least 8 and containing from about 4 percent to 10 percent cellulose, from about 4 percent to 11 percent caustic soda, from about 35 percent to 50 percent carbon disulfide based on the weight of the cellulose and from about 0.5 percent to 3 percent, based on the weight of the cellulose, of an polyxylalkylene glycol ether of a substance selected from the group consisting of castor oil, hydrogenated castor oil and mixtures of castor oil and hydrogenated castor oil into an aqueous spinning bath containing from about 10 percent to 25 percent sodium sulfate, from about 4 percent to 12 percent zinc sulfate and sulfuric acid, the sulfuric acid content of the spinning bath exceeding the slubbing point but not exceeding about 9 percent, removing the body from the spinning bath, washing the body and drying the body.

4. The steps in the method as defined in claim 3 wherein the shaped bodies consist of filaments and the ether is a polyoxyethylene glycol ether of castor oil containing from about 30 to 100 ethylene oxide units per molecule.

5. The method of producing shaped bodies of regenerated cellulose consisting substantially entirely of skin which comprises adding to and incorporating in a viscose from about 0.5 percent to 2 percent, based on the cellulose in said viscose, of an alkali-soluble polyoxyalkylene glycol ether of a substance selected from the group consisting of castor oil, hydrogenated castor oil and mixtures of castor oil and hydrogenated castor oil, the viscose containing from about 4 percent to 10 percent cellulose, from about 4 percent to 11 percent caustic soda and from about 35 percent to 50 percent carbon disulfide, the percentage of said carbon disulfide being based upon the weight of the cellulose, and extruding the viscose at a salt index of at least 8 into an aqueous spinning bath containing from about 10 percent to 25 percent sodium sulfate, from about 4 percent to 15 percent zinc sulfate and sulfuric acid, the percentage sulfuric acid content of the bath exceeding the slubbing point but not exceeding about 0.2 times the percentage carbon disulfide content of the viscose, based on the weight of the cellulose in the viscose.

6. The method as defined in claim 5 wherein the ether is a polyoxyethylene glycol ether of castor oil containing from about 30 to 100 ethylene oxide units per molecule.

7. The method of producing shaped bodies of regenerated cellulose consisting substantially entirely of skin which comprises adding to and incorporating in a viscose

from about 0.5 percent to 2 percent, based on the weight of the cellulose in said viscose, of an alkali-soluble polyoxyalkylene glycol ether of a substance selected from the group consisting of castor oil, hydrogenated castor oil and mixtures of castor oil and hydrogenated castor oil, the viscose containing about 7 percent cellulose, about 6 percent caustic soda and about 41 percent carbon disulfide, said percent carbon disulfide being based upon the weight of the cellulose, ripening the viscose to a salt point of not less than about 9 and extruding the viscose into an aqueous spinning bath containing from about 15 percent to 22 percent sodium sulfate, from about 5 percent to 12 percent zinc sulfate and sulfuric acid, the sulfuric acid content of the spinning bath exceeding the slubbing point but not exceeding about 9 percent.

8. The method as defined in claim 7 wherein the ether is a polyoxyethylene glycol ether containing from about 20 to 200 ethylene oxide units per molecule.

9. The method as defined in claim 7 wherein the ether is a polyoxyethylene glycol ether of castor oil containing from about 30 to 100 ethylene oxide units per molecule.

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