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TEXTILE TREATMENT PROCESS

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This invention relates to textiles and more particularly to a new process for the treatment of textiles to render them water repellent.

Unmodified synthetic or natural textile fibers as a whole are not water repellent, that is, they are readily wet out by water. It has been known for many years that treatment of fabric or yarns with waxy materials will render them water repellent but the finishes produced by the older treatments in general have all suffered from lack of resistance to laundering or dry cleaning. In recent years a number of water repellent agents and treatments have been developed which give water repellent effects which are resistant to laundering. Most of these are based on expensive chemicals or complex treatments.

This invention has as an object the provision of a process for rendering textiles water repellent which is simple and involves the use of readily available low-cost chemicals to give water repellent effects which are durable toward laundering. A further object comprises the textiles thus produced. Other objects will appear hereinafter.

These objects are accomplished by the following invention wherein a textile material is rendered water repellent by treatment with a monofunctional acylating agent in combination with a monomeric, non-polymerizable, substantially water-insoluble amine containing at least ten carbon atoms and not more than two amino groups, in which amine the nitrogen atom of at least one amino group bears at least one hydrogen atom, and baking the treated textile material to complete the reaction, i. e. until the textile material is rendered water repellent.

It is believed that the water repellency produced by the present invention is achieved by forming a long chain hydrophobic amide in situ in the fabric. The high degree of laundry fastness is apparently connected with the formation of the amide deep within the fiber which makes the hydrophobic material very difficult to remove.

In its preferred form the process of the invention is carried out by first impregnating the fabric with the amine in the form of a water-soluble salt following which the acylating agent is applied and the fabric heated to effect reaction. The amine can be introduced into the fabric by impregnation followed by squeezing with pressure rolls or by centrifugal wringing or it may be sprayed into the fabric at a controlled rate using a fine spray. It may be applied either as a solution or in liquid form. The use of a solution is advantageous since it permits the application of the very small amount of reagent which it has been found is sufficient to give the desired degree of water repellence. The fabric impregnated with amine is preferably dried before application of the acylating agent. This is particularly desirable when the amine has been introduced into the fabric in the form of an amine salt from an aqueous solution or where the solvent employed is one which reacts with acylating agent, for example, alcohol. The acylating agent is usually applied from a solution in an unreactive solvent, for example, toluene, benzene, or carbon tetrachloride. This minimizes loss of reagent due to side reactions. The acylating agent may also be applied in undiluted form but in general this requires excess reagent which is wasteful. The excess acylating agent or acylating solution is then removed by wringing or centrifuging or squeezing if desired after which the treated material is either dried in a current of air at room temperature and heated or is heated without preliminary drying to effect reaction. In general, the treatment may be carried out on equipment which is well known in the textile art and no description of such apparatus is required here.

The more detailed practice of the invention is illustrated by the following examples, wherein parts given are by weight. There are of course many forms of the invention other than these specific embodiments.

Example I

This example illustrates a preferred method of introducing the amine component into the fabric.

Fifty-four parts of octadecylamine hydrochloride is dissolved in 72,000 parts of water. The solution is heated to boiling following which 2500 parts of mercized unfinished broadcloth is introduced into the solution after which heating is continued for 30 minutes. The fabric is removed, centrifuged to twice the weight of the dry fabric and dried. The fabric has increased in weight about 0.8%, indicating an appreciable concentration of the octadecylamine hydrochloride in the fabric.

Example II

This example illustrates the production of permanent water repellence using a combination of a long chain amine hydrochloride with a long chain anhydride.

A portion of the fabric prepared in Example I is steeped in a 5% solution of stearic anhydride in ethylene chloride. The fabric is removed, wrung to twice the weight of the dry fabric and heated for 30 minutes at 120° C. The treated fabric, following the baking, possesses a high degree of water repellency. The repellency is not reduced by sudsing with 0.2% of warm mild soap solution followed by water or by boiling with soap and soda ash for two hours.

A piece of similar fabric which contained no octadecylamine hydrochloride is treated with

stearic anhydride as described in the previous paragraph. The degree of water repellency obtained is markedly lower.

Example III

This example illustrates the treatment of cotton broadcloth with a long chain amine salt in combination with an acylethenone.

A portion of the fabric prepared in Example I is impregnated with a 5% solution of octadecanoyhexadecylethenone. The fabric is wrung, dried at room temperature, and then cut into two pieces and heated as follows:

Sample 1---------- 20 minutes at 150° C.
Sample 2---------- 30 minutes at 150° C.

The fabrics after sudsing in mild soap at 160° F. followed by thorough rinsing to remove soap possess a high degree of water repellency. After boiling for two hours with a mixture of soap and soda ash, the sample baked for 20 minutes at 150° C. is only very slightly lowered in repellency while the sample baked for 30 minutes at 150° C. is unchanged.

Example IV

This example illustrates the use of a high carbon content aromatic amine in conjunction with a long chain aliphatic anhydride.

Two parts of benzidine, ten parts of glacial acetic acid, and 96 parts of water are warmed together until the benzidine dissolves. Six pieces of broadcloth are steeped in this solution for ten minutes following which they are wrung to twice the weight of the original fabric and dried. The fabrics are then steeped in a mixture composed of three parts of stearic anhydride and 97 parts of ethylene chloride for five minutes after which they are wrung to twice the weight of the original fabric and dried. Three samples are baked by heating for 20 minutes at 150°, five minutes at 170°, 20 minutes at 170°, respectively. The remaining samples are baked at times ranging from 20 to 60 minutes at 120°. All the treated fabrics are rinsed thoroughly in mild soap at 160° F. for five minutes. The products except the one baked at 120° for 20 minutes possess a high degree of water repellency. After laundering by boiling for two hours with soap and soda ash, the fabrics baked at 120° are moderately lower in water repellency while those baked at 150–170° are substantially unchanged. After a second laundering of the same type, the water repellency of the samples is substantially unchanged. Apparently, at lower reaction temperatures, the degree of reaction obtained is not as great as that produced at the higher temperatures but the degree of laundry-fastness is essentially the same.

Example V

This example illustrates the treatment of cotton broadcloth using benzidine hydrochloride as the amine component and stearic anhydride as the long chain monofunctional acylating agent.

Six pieces of mercerized cotton broadcloth are steeped in a solution of two parts of benzidine in a solution of two parts of concentrated hydrochloric acid in 96 parts of water for five minutes after which they are wrung to twice the weight of the dry fabric and dried. The fabrics are then steeped in a solution of three parts of stearic anhydride and 97 parts of ethylene chloride for five minutes, after which they are wrung to a weight equal to twice the weight of the original dry fabric, dried at room temperature and baked for different lengths of time, varying from 20 to 60 minutes at 120° C. and 5 to 20 minutes at 170° C. The fabrics are then given a quick laundering in dilute mild soap solution at 160° F. All the fabrics are strongly water repellent. The fabrics are then boiled for two hours in soap and soda ash solution. The repellency is essentially unchanged. Even after another vigorous laundering of this type, the repellency is still of the same order as that of the unlaundered treated fabric.

Example VI

This example illustrates the production of a water repellent effect on cotton jean cloth by treatment with a short chain acylating agent (butyric anhydride) in combination with stearylamine hydrochloride.

Cotton jean cloth is boiled for 30 minutes in an 0.07% aqueous solution of stearylamine hydrochloride. The cloth is then removed and dried thoroughly. The cloth is next treated with a 5% dry benzene solution of butyric anhydride after which it is heated at 120° C. for 20 minutes. The cloth is resistant to wetting by water. The repellency is not reduced by thorough laundering.

Example VII

This example illustrates the treatment of cotton jean cloth to render it water repellent using a combination of lauric anhydride and octadecylamine hydrochloride.

Cotton jean cloth treated as in Example I is impregnated with a 5% dry benzene solution of lauric anhydride. The excess solution is removed by wringing after which the fabric is dried at room temperature and cured by heating at 120° C. for 20 minutes. The treated fabric after rinsing with warm mild soap and water is moderately water repellent. After laundering thoroughly the water repellency is somewhat improved apparently because of the removal of surface active by-products.

Example VIII

This example illustrates the use of hexyl isocyanate combined with octadecylamine hydrochloride on cotton jean cloth to render it water repellent.

Cotton jean cloth impregnated with octadecylamine hydrochloride as described in Example VI is treated with a 5% dry benzene solution of hexyl isocyanate. The excess treating solution is removed, the fabric dried at room temperature and cured at 120° C. for 20 minutes. The fabric is rinsed with warm soap and water and then laundered. The fabric is water repellent even after the laundering.

Example IX

This example illustrates the use of octadecylamine hydrochloride in combination with octadecylphenyl isocyanate on cotton jean cloth to render it water repellent.

Cotton jean cloth is treated with octadecylamine hydrochloride as described in Example VI. The dry cloth is then impregnated with a 5% dry benzene solution of octadecylphenyl isocyanate. The excess treating solution is removed following which the fabric is heated at 120° C. for 20 minutes. The fabric after rinsing with warm soap and water shows moderate water repellency. After thorough laundering to remove by-products which tend to reduce water repellency the fabric is highly water repellent.

Example X

This example illustrates the treatment of cotton jean cloth with octadecylamine hydrochloride in combination with phenyl isocyanate to produce a water repellent effect.

Cotton jean cloth is impregnated with octadecylamine hydrochloride as described in Example VI. The dry fabric is then treated with a 5% dry benzene solution of phenyl isocyanate. The excess impregnating solution is removed, the fabric dried and heated at 120° C. for 20 minutes. The fabric after rinsing with warm soap and water is of good water repellency. The repellency is not reduced by a thorough laundering.

Example XI

This example illustrates the treatment of cotton jean cloth containing no octadecylamine hydrochloride with butyric anhydride and shows that the water repellency is improved by the presence of the amine salt.

Cotton jean cloth containing no octadecylamine hydrochloride is impregnated with a 5% dry benzene solution of butyric anhydride. The excess treating solution is removed and the fabric dried at room temperature. It is then heated at 120° C. for 20 minutes. After rinsing with warm soap and water the fabric shows no water repellency whatsoever. After thorough laundering to insure removal of interfering substances, there is still no indication of water repellency.

While the process of the present invention is of particular value in its application to cotton fabrics and this application represents a preferred phase of the invention, the process may be applied to all types of organic fibrous materials and is therefore generically applicable to the treatment of such materials as cotton, linen, leather, silk, wool, jute, hemp, coir, sisal, paper, hair, ramie, flax, alpaca, wood pulp and the like.

In carrying out the treatment on fibers such as viscose rayon, rayon staple, acetate rayon, silk, wool, and synthetic protein fibers, the process described in Example II may be used with advantage. The fibrous material may be in the raw state or in any processed state in which its fibrous nature is retained such as wood pulp or in the form of a manufacture such as cord, thread, fibers, paper, belts, fabrics, and especially textiles. Particularly significant results are obtained with cellulosic materials, that is, materials having a substituted or unsubstituted cellulosic nucleus including linen, cotton, cellulose acetate rayon, viscose rayon, rayon staple, and cuprammonium rayon. The cellulose nucleus preferably should be unsubstituted as is the case with cellulosic fabrics made from cotton, linen, and viscose and cuprammonium rayons. The best results are also obtained when the treated material is free of extraneous finishing material such as starch, gum, etc.

The water-insoluble amines which are applicable for the treatment are those which contain at least one free amino hydrogen. The amines are further characterized by the fact that they contain at least ten carbon atoms preferably in one uninterrupted hydrocarbon chain attached directly to the amino nitrogen. The amines are further characterized by the fact that they are not film-forming, that is, they are not of a sufficiently high molecular weight to show resinous characteristics. The non-resinous state may be taken roughly as including amines of molecular weight up to 750. This is important since only non-resinous amines are capable of penetration into the fiber to an extent sufficient to give the deep-seated degree of repellency produced by the present process. Amines of film-forming type such as those produced by the deacetylation of chitin give good repellency but their action is restricted essentially to the insolubilization of a surface coating. The amines of this invention preferably contain an aliphatic hydrocarbon chain of at least ten carbon atoms as one of the substituents on the nitrogen atom. However, those amines derived from other radicals are also effective, particularly when the amine is one which is substantive toward the fiber being treated. Examples of such materials are salts of amines of the benzidine series and salts of long chain aliphatic amines.

In the process of this invention any monomeric, amide-forming, substantially water-insoluble amine which is resistant to polymerization and which contains not more than two amino groups, neither of which is tertiary, at least ten carbon atoms and at least one hydrogen on amino nitrogen may be used including phenylethylbutylamine $C_6H_5CH_2CH_2NHC_4H_9$, stearylamine, oleylamine, dicetylamine, dioctylamine, dicyclohexylamine, laurylamine, N-methylstearylamine, 2-aminodecane, benzidine, and tetrahydrofurfuryllaurylamine. A special group of amines suitable for the reaction are the monoacylated diamines, e. g. 2-stearamidoethylamine, 4-stearamidobutylamine, 3-oleylamidopropylamine, etc. The amine may be used either in the form of its water-soluble salts or may be applied from an organic solvent solution. Use of the amine in the form of its salts constitutes a preferred embodiment of the present invention since in general amine salts of this type tend to show substantivity toward cellulose which in turn tends to improve the laundry resistance of the finished treated fabric.

Tertiary amines are not suitable for this process. Likewise amines derived from short chain radicals even though they be primary or secondary are not appropriate. Thus, for example, compounds such as diethylenetriamine or triethylenetetramine do not give worthwhile degrees of repellency according to the process of the invention. The amine in free form, that is not in the form of its salts, should be substantially water-insoluble since it has been found that water-soluble amines in general give products of inferior water repellency. For the purposes of this invention amines showing a greater solubility than 2% in water at 25° C. are considered as water-soluble.

Any monofunctional acylating agent may be used in the process of the present invention, but those acylating agents containing radicals of at least four carbon atoms are preferred. As acylating agents there may be employed the isocyanates, the isothiocyanates, the anhydrides derived from carboxylic acids, both aromatic and aliphatic, anhydrides derived from sulfonic acids, ethenones including the acyl substituted ethenones, acyl isothiocyanates, acyl isocyanates, acid halides, acid azides, and mixed anhydrides. As a rule, however, the acid chlorides are less advantageous in spite of the fact that they give very good repellency since they cause appreciable tendering of the fabric unless the reaction is carried out under exceedingly mild conditions. The acylating agent employed for the reaction is most preferably a long chain aliphatic acid anhydride such as those derived from aliphatic acids containing eight or more carbon atoms.

Best water repellency is obtained with aliphatic acid anhydrides preferably saturated derived from acids containing at least twelve carbon atoms. Acylating agents containing other groups, however, may be employed, for example, aromatic isocyanates, heterocyclic isocyanates, or anhydrides and anhydrides containing substituent groups. For good water repellency the number of carbon atoms in the amine plus the number of carbon atoms of the acylating agent should ordinarily equal at least 20.

In making such calculation in the case of acid anhydrides the number of carbon atoms of the acylating agent should be taken as half the total number of carbon atoms of the anhydride. This method of calculation may also be employed in the case of mixed anhydrides. It is assumed in the case of mixed anhydrides that considering the reaction mixture as a whole both types of acyl groups will be involved in the reaction although of course only one of the acyl groups of any given mixed anhydride molecule will react.

Specific acylating agents which illustrate the generic invention are acetic anhydride, butyric anhydride, valeric anhydride, caproic anhydride, capric anhydride, lauric anhydride, stearic anhydride, arachidic anhydride, hexyl isocyanate, phenyl isocyanate, lauryl isocyanate, and octadecyl isocyanate, octadecyl isothiocyanate, stearoyl isothiocyanate, stearoyl isocyanate, stearic acid chloride, butyric acid chloride, p-toluenesulfonic acid chloride, p-toluenesulfonic anhydride, benzoic anhydride, benzoyl chloride, furoic acid chloride, octadecyl chlorocarbonate, and p-nitrophenyl isocyanate. The acylating agent may be saturated or unsaturated in the alkyl or acyl chain and may be straight or branched chain and substituted or not by halogen, ether, ketone, ester, sulfide, nitrile, and other groups. For maximum water repellency, however, the latter and other non-hydrocarbon groups should be avoided. Mixtures of acylating agents as well as mixtures of amines may be employed.

The agents may be used in conjunction with other treating agents or processes. Suitable effective combinations may be produced with resins, waxes, softening agents, sizes, formaldehyde, dimethylolurea, and the like. Good effects are obtained when treatments are applied simultaneously, but preferably consecutively in creaseproofing processes. While it is not essential to do so, the acylating agents are preferably applied to the fabric from an organic solvent solution which offers the advantage of excluding moisture and giving uniform treatment with an adequate amount of the acylating agent. In general, any liquid (under the conditions of the process) which is a solvent for and chemically inert toward the acylating agent may be used. Suitable specific solvents include aliphatic hydrocarbons, for example, petroleum naphtha, carbon tetrachloride, trichloroethylene, tetrachloroethylene, ethylene dichloride, benzene, toluene, dioxane, diethyl ether, 1,3-dichlorobenzene, ethyl acetate, acetone, methyl ethyl ketone, and diisopropyl ether. The use of a solvent minimizes the possibility of undesirable side reactions. The acylating agent, however, may also be applied from aqueous emulsions when it contains a long chain, for example, 12 or more carbon atoms. Although these reagents react slowly with the water in aqueous emulsions they may be preserved in contact therewith for reasonable lengths of time. The application of the shorter chain acylating agents from aqueous emulsions, while in some instances possible if manipulation is rapid, is not recommended because they are more and more sensitive to water as the chain length decreases. If the acylating agent is a solid, it may be melted and the fibrous body previously impregnated with the amine immersed in the melt. Similarly, if the acylating agent is normally a liquid, the fibrous substance may be steeped therein in the absence of a solvent and even heated in this condition, thus avoiding the necessity to remove the acylating agent before curing. These methods are not preferred, however, since the results are not always uniform and, in general, they are more expensive than those using solvents.

The quantity of the acylating agent that may be applied to the fibrous material is easily determined by the ability of the fibers to absorb liquid. Thus, where the fibers are in loose form, a weight of solution equal to as much as three times the weight of the material is easily absorbed. On the contrary, if the fiber surface is compact, the fibrous body may absorb only a small fraction of its weight. This, however, is not material since detectable effects are obtained with amounts of acylating agent and amine as low as 0.5%, the two reagents being employed in substantially stoichiometric amounts. On the other hand, as much as 20% or more of the combination may be applied by proper regulation of the concentration. The quantity of acylating agent employed in no case should be sufficiently great to impair the fibrous structure of the material under treatment. It is usually desirable to employ excess acylating agent over the stoichiometric quantity required for reaction with the amine. Following application of the amine or the acylating agent, the excess treating solution is preferably removed. This is most conveniently accomplished by wringing, centrifuging, or any similar procedure. The residual solvent or dispersion medium may next be and usually is removed by evaporation. Where an aqueous emulsion of the acylating agent has been employed, it is usually advantageous to remove the water at a temperature below 60° C. in order to prevent or minimize reaction between the water and the acylating agent during the drying step. However, preliminary drying is not necessary when the agent has been applied from an aqueous emulsion if the baking treatment is applied under such conditions that the fabric can be dried rapidly at an elevated temperature. Where drying at elevated temperature takes place rapidly (five minutes or less), little reaction with the water seems to occur during this step. When the acylating agent has been applied from an organic solvent solution, the conditions which may be used for drying need not be so carefully controlled. Fire hazards are minimized by drying at low temperatures and for this reason drying temperatures of 60–100° C. are recommended in the case of organic solvents.

Following application of the acylating agent and drying, the fabric is baked or cured to effect reaction. Temperatures of about 70 to 150° C. are in general satisfactory for this purpose. At such temperatures, the agent is usually highly effective and the fibers do not suffer serious degradation. However, temperatures of about 90 to 135° C. are preferred because degradation at these temperatures is negligible unless inorganic acid is present such as may be produced from an acid chloride. In some instances, as where the agent is rather inert, temperatures above 150° C. are necessary to effect reaction although baking above 200° C. should nearly

always be avoided unless the reaction can be carried out with extreme rapidity. The time of heating depends upon the activity of the particular acylating agent and amine employed. Times of reaction up to 30 minutes have been found satisfactory. Times of reaction up to one hour are permissible with agents which react slowly. The heating should always be carried out within such intervals as are not too great as to harm the fibrous materials seriously, that is, impair its fibrous structure. This is at most a matter of simple test. The temperature chosen for the reaction therefore in general depends primarily upon the reactivity of the agents employed. In some instances where the reagent is very reactive, the reaction may be carried out at room temperature without heating or by very gentle heating to temperatures not exceeding 60°. In the case of acid chlorides such a procedure is virtually essential in order to achieve repellency without serious degradation. If desired the acylating agent can be applied prior to the amine or even simultaneously with the amine if care is taken to minimize reaction with the amine prior to application to the textile material.

The process is primarily applicable to the production of durable water repellent textile materials.

The above description and examples are intended to be illustrative only. Any modification of or variation therefrom which conforms to the spirit of the invention is intended to be included within the scope of the claims.

What is claimed is:

1. A textile of improved water repellence having impregnated therein the amide reaction product, formed in situ, of a monofunctional acylating agent of the class consisting of organic isocyanates, isothiocyanates, carboxylic acid anhydrides, sulfonic acid anhydrides, ethenones, acyl halides and acyl azides with a monomeric, non-polymerizable, substantially water-insoluble amine containing not more than two amino groups, neither of which is tertiary, at least one hydrogen on amino nitrogen, and at least ten carbon atoms.

2. A cellulosic textile of improved water repellence having impregnated therein the amide reaction product, formed in situ, of a monofunctional acylating agent of the class consisting of organic isocyanates, isothiocyanates, carboxylic acid anhydrides, sulfonic acid anhydrides, ethenones, acyl halides and acyl azides with a monomeric, non-polymerizable, substantially water-insoluble amine containing not more than two amino groups, at least one hydrogen on amino nitrogen, and at least ten carbon atoms.

3. A cotton textile of improved water repellence having impregnated therein the amide reaction product, formed in situ, of a monofunctional acylating agent of the class consisting of organic isocyanates, isothiocyanates, carboxylic acid anhydrides, sulfonic acid anhydrides, ethenones, acyl halides and acyl azides with a monomeric, non-polymerizable, substantially water-insoluble amine containing not more than two amino groups, at least one hydrogen on amino nitrogen, and at least ten carbon atoms.

4. A cellulosic textile of improved water repellence having impregnated therein the amide reaction product, formed in situ, of a monofunctional acylating agent of the class consisting of organic isocyanates, isothiocyanates, carboxylic acid anhydrides, sulfonic acid anhydrides, ethenones, acyl halides and acyl azides with a monomeric, non-polymerizable, substantially water-insoluble amine containing not more than two amino groups, at least one hydrogen on amino nitrogen, and at least ten carbon atoms, and substantive, in the form of its salts, for cellulose.

5. A cellulosic textile of improved water repellence having impregnated therein the amide reaction product, formed in situ, of a monofunctional acylating agent of at least four carbon atoms of the class consisting of organic isocyanates, isothiocyanates, carboxylic acid anhydrides, sulfonic acid anhydrides, ethenones, acyl halides and acyl azides with a monomeric, non-polymerizable, substantially water-insoluble amine containing not more than two amino groups, at least one hydrogen on amino nitrogen, and at least ten carbon atoms.

6. A cellulosic textile of improved water repellence having impregnated therein the amide reaction product, formed in situ, of a saturated aliphatic carboxylic acid anhydride and a monomeric, non-polymerizable, substantially water-insoluble amine containing not more than two amino groups, at least one hydrogen on amino nitrogen, and at least ten carbon atoms.

7. A cellulosic textile of improved water repellence having impregnated therein the amide reaction product, formed in situ, of a saturated aliphatic carboxylic acid anhydride, derived from an acid of at least twelve carbon atoms, and a monomeric, nonpolymerizable, substantially water-insoluble amine containing from one to two amino groups, at least one hydrogen on amino nitrogen, and at least ten carbon atoms.

8. Process which comprises impregnating a textile with at least 0.5% of a monofunctional acylating agent of the class consisting of organic isocyanates, isothiocyanates, carboxylic acid anhydrides, sulfonic acid anhydrides, ethenones, acyl halides and acyl azides and at least 0.5% of a monomeric, non-polymerizable, substantially water-insoluble amine containing not more than two amino nitrogens, at least ten carbon atoms and at least one hydrogen on amino nitrogen and curing the fabric impregnated with the amine and the acylating agent at 70–150° C.

9. Process of claim 8 wherein the textile is cellulosic and the amine is substantive to cellulose.

10. Process of claim 8 wherein the acylating agent contains at least four carbon atoms.

11. Process of claim 8 wherein the acylating agent is a saturated aliphatic carboxylic acid anhydride.

12. Process of claim 8 wherein the acylating agent is a saturated aliphatic carboxylic acid anhydride derived from an acid of at least twelve carbon atoms.

13. Process of claim 8 wherein the acylating agent is a saturated aliphatic isocyanate of at least four carbon atoms.

14. Process of claim 8 wherein the sum of the reaction residues of the amine and acylating agent contains at least twenty carbon atoms.

15. Process of claim 8 wherein the amine contains an aliphatic hydrocarbon chain of at least ten carbon atoms directly attached to the amino nitrogen.

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