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TEXTILE FINISHING

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16 Claims. (Cl. 260—29)

This invention relates to the treating and finishing of textiles and textile-forming materials. The invention includes processes for finishing textiles, textile finishing compositions, the preparation of such textile finishing compositions, and textiles of improved properties finished with the compositions and by the processes of the invention.

Textile fabrics, yarns, threads and fibers have been heretofore treated with a wide variety of creaseproofing agents, waterproofing agents, materials to prevent shrinking, felting, fulling, etc., lubricants, softening agents, sizes, binding agents and other materials to improve the appearance and "feel" or hand of the goods. The treatment of textiles for these and similar purposes is known as textile finishing, and the agents employed for this purpose are known as textile finishing agents.

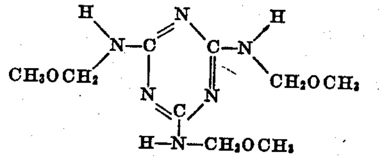
It is a principal object of the present invention to provide textile finishing compositions and methods for their preparation and application to textiles, which compositions and methods will produce finished textiles having a greatly improved water resistance, a more desirable hand, crease resistance, a very much reduced tendency for shrinking and other desirable improvements which will be apparent from the description which follows: Other important objects will appear hereinafter.

The various textile materials which may be treated in accordance with my invention include fibers, thread, yarns, knit and woven cloth and other fabricated materials of cotton, linen, hemp, jute, regenerated cellulose, cellulose esters, cellulose ethers, wool, synthetic wool, silk, synthetic silk and other fibrous materials whether natural or synthesized. Hereinafter and in the claims for purposes of brevity all of these various textile-forming materials and structures containing the same will be referred to as textile fabrics.

Although textile fabrics having a greatly reduced tendency to shrink as well as crease resistance and an improved hand may be obtained by treatment with aqueous dispersions of alkylated methylol melamine followed by drying and curing the resin in the fabric, fabrics so treated have but a fair resistance to the penetration of water and therefore leave much to be desired insofar as their water-repellent characteristics are concerned. By treatment of textile fabrics with compositions such as will be presently described I am enabled to retain all of the advantages of shrink-resistance, crease-resistance, improved hand, etc. which are obtainable by treatment of the fabric with lower alkylated methylol melamines and in addition impart to such fabrics a very high degree of water-repellency.

My improved textile finishing process includes the steps of treating textile fabrics such as mentioned above with dispersions containing alkylated methylol melamine together with certain nitrogen-containing compounds such as will be presently described, followed by drying and curing these resin-forming constituents in the fabric. The improved results which I obtain are due, I believe, to certain chemical reactions which take place during the drying and curing treatment between the alkylated methylol melamine and the nitrogen-containing compound employed therewith.

When a textile fabric treated with an alkylated methylol melamine, for example methylated methylol melamine, having a structure such as

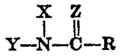


is subjected to a heat treatment the methylated methylol melamine polymerizes, or cures, in the fabric. Curing of the resin probably takes place by splitting off one or more molecules of methyl alcohol followed by a molecular rearrangement which favors polymerization. While, as stated before, fabrics so treated have a very marked resistance to shrinking, a good hand, crease-resistance and other important characteristics the fabrics are not particularly water-repellent. This lack of water resistance is due in part at least to the fact that the polymerized resin molecule has no effective hydrophobic groups available to repel water. In other words, the cured resin is fairly easily wetted and water is allowed to penetrate into the interstices of the fabric.

By adding to the alkylated methylol melamine textile treating composition a nitrogen-containing compound of the class to be presently described and then impregnating, drying and curing the fabric at relatively low temperatures, a chemical reaction is believed to take place between the alkylated methylol melamine and the nitrogen-containing compound which modifies the nature of the cured resin but does not materially influence the polymerization mechanism necessary for obtaining a permanent water-insoluble cured resin in the fabric.

In my copending application Serial No. 409,112, filed August 30, 1941, I demonstrated that guanamines having a hydrophobic alkyl radical of at least 7 carbon atoms and aldehyde condensation products of such guanamines will produce a high degree of water-repellency on textile fibers when employed with alkylated methylol melamine. My present invention in its broader aspects is based on the discovery that any compound having a hydrophobic alkyl radical of at least 7 carbon atoms and containing a nitrogen atom having attached thereto an acidic group can be employed in conjunction with alkylated methylol melamines for textile finishing. Combination between the compound containing the acidic nitrogen atom, which is designated herein as an amido group, and the alkylated methylol melamine is brought about under the conditions hereinafter described by means of a reactive hydrogen present on the amido group or on a substituent such as an alkylol radical attached thereto. Although other substituents may also be present on the amido nitrogen the compound as a whole should be free from basic salt-forming groups, since I have found that the waterproofing effect obtained upon fabrics is not permanent when such salt-forming groups are present. This lack of permanency is perhaps due to the water-solubility of the salts so formed and it is an advantage of my invention that most of the compounds which I employ are acidic in character and do not easily form salts.

Although my invention in its broader aspects is substantially as outlined above, the preferred nitrogen-containing compounds of my invention are those of the formula



$$Y-\overset{\overset{\textstyle X}{|}}{N}-\overset{\overset{\textstyle Z}{\|}}{C}-R$$

in which Y is a member of the group consisting of hydrogen and unsubstituted or substituted alkylol radicals, such as methylol or ethylol radicals, Z is a member of the group consisting of oxygen and sulfur, and X and R are members of the group consisting of hydrogen, non-functional substituents, and radicals containing alkyl hydrophobe groups of at least 7 carbon atoms, it being understood that in all cases at least one X or R contains a hydrophobe alkyl radical of at least 7 carbon atoms. The mechanism of the condensation between methylated methylol melamine and the amido compounds of this class will now be explained.

I have found that amides of the type represented by the formula $H_2N.CO.R'$ in which R′ is a higher alkyl radical, can be condensed with methylated methylol melamines by heating the mixture on the textile fibers to be treated, preferably in the presence of an acid catalyst. The reaction is probably as follows:

$$M—NH.CH_2O.CH_3+H.NH.CO.R' \rightarrow M—NH.CH_2.NH.CO.R'+CH_3OH$$

wherein M is the residue of methylated methylol melamine and R′ is a higher alkyl radical.

A similar reaction can be carried out on the fibers between methylated methylol melamine and an aldehyde condensation product of an amide. It is known that aldehydes such as formaldehyde will combine with amides to form methylol derivatives by the reaction:

$$O:CH_2+H_2N.CO.R' \rightarrow HO.CH_2NH.CO.R'$$

The reaction between these compounds and the methylated methylol melamines is probably as follows:

$$M—NH.CH_2.O.CH_3+HO.CH_2.NH.CO.R' \rightarrow M—NH.CH_2.O.CH_2.NH.CO.R'+CH_3OH$$

Another class of derivatives of aliphatic amides which can be reacted with methylated methylol melamines on the fiber are those corresponding to the formula $HO.(CR^2R^3)_n.NH.COR'$, where R^2 and R^3 are hydrogen or alkyl groups and n is a whole number greater than one. These compounds are easily prepared by reacting an alkylolamine with a higher fatty acid to form an amide thereof. Thus, for example, ethanolamine or propanolamine heated with stearic acid will form amides of the formula:

$$HO(CH_2)_n.NH.CO.C_{18}H_{37}$$

wherein $n=2$ or 3. These amides will condense with methylated methylol melamine on the textile fibers in the presence of an acid catalyst, the reaction probably being as follows:

$$M—NH.CH_2O.CH_3+HO(CH_2)_n.NH.CO.C_{18}H_{37} \rightarrow M—NH.CH_2.O(CH_2)_n.NH.CO.C_{18}H_{37}+CH_3OH$$

From the foregoing it is evident that all the preferred nitrogen-containing compounds defined above will condense with methylated methylol melamine by splitting out methanol. The primary condensation products are therefore exemplified by the general formula:

$$M—NH.G.\overset{\overset{\textstyle X}{|}}{N}.\overset{\overset{\textstyle Z}{\|}}{C}.R$$

where G is a coupling linkage of the group $—CH_2—$ and $—CH_2O(CR^2R^3)_n—$, R^2 and R^3 being members of the group consisting of hydrogen and alkyl radicals; n is a small whole number, and M, X, Z and R are as previously defined.

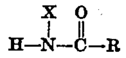
In these condensation products at least one of the short chain groups of the methylated methylol melamine is replaced with a group containing in X or R, or both, one or more long chain hydrophobic alkyl groups. It will be noted that the product also contains in the melamine nucleus at least one methoxy methyl group which at the temperature of the curing process can split off methanol, allowing a molecular rearrangement to take place with subsequent polymerization with other alkylated methylol melamine molecules. The amido compound having the long chain alkyl group which replaces the short chain alkyl group is not split off at the temperature of the curing process and hence the long chain alkyl radical remains as a part of the resin in which it acts to render the resin itself hydrophobic.

It is to be clearly understood that the above discussion is merely a suggested mechanism of what takes place in my finishing process, and I do not intend to be bound thereby since regardless of whatever theory may be advanced the superior results obtained by my process remain as a distinct advance in the art.

It should also be understood that my invention in its broader aspects is not limited to the use of methylated methylol melamines as illustrated above. Alkylated methylol melamines having short alkyl groups of not more than four carbon atoms such as ethylated, isopropylated and butylated methylol melamines may also be employed, but require greater care in the curing steps. Ethyl alcohol and butyl alcohol split off during the curing steps with greater difficulty and a proper cure is more difficult to obtain without employing temperatures which might damage the fabric.

The alkylated methylol melamines which I employ are prepared by known methods. Methylated methylol melamine may, for example, be prepared by reacting 2 to 6 moles of formaldehyde with 1 mole of melamine to form a condensation product believed to be mostly methylol melamine. This product is then reacted with 2 to 6 moles of methanol whereby methylated methylol melamine is formed. By the term methylated methylol melamine I intend to include all of these various reaction products containing from 2 to 6 methylated methylol groups per molecule. Excessive polymerization should be avoided during the preparation since the resin is preferably employed by me in the form of an aqueous dispersion, or solution, and products polymerized excessively tend to be difficultly soluble in water. I have also found that excessive polymerization of the product tends to give the fabrics a stiff hand.

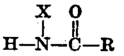
A very large number of nitrogen-containing compounds having the characteristics set forth above are suitable for use with alkylated methylol melamines in my textile finishing composition. The preferred class of compounds of this type are for example, the amides of monocarboxylic acids which, in the general formula



$$H-\overset{X}{\overset{|}{N}}-\overset{O}{\overset{\|}{C}}-R$$

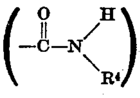
X is hydrogen and R is an alkyl radical of at least seven carbon atoms, such as, for example, caprylamide, capramide, lauramide, myristamide, palmitamide, stearamide, di-n-octyl-acetamide, oleic acid amide, linoleic acid amide and ricinoleic acid amide. Other amides of the same general formula, but in which X is an alkyl group, may be employed. Such compounds are, for example, N-methylstearamide, N-ethylstearamide, N-ethyllauramide, N-isopropylstearamide, N-2-ethylhexylstearamide and the like. Amides of the general formula above in which H is replaced with an alkylol group such as methylol stearamide, methylol lauramide, ethanol stearamide and others of this class may also be employed.

Compounds may also be employed having the general formula



$$H-\overset{X}{\overset{|}{N}}-\overset{O}{\overset{\|}{C}}-R$$

in which X is a cyano radical as for example, lauroyl cyanamide, stearoyl cyanamide, or an alkylene nitrile ($-CR'R^2C\equiv N$) radical when using compounds such as, di(octyl)cyanacetamide, di(lauryl)cyanacetamide, di(stearyl) cyanacetamide, N-β-cyanethyllauramide, etc.; or a carbamyl radical, as in the case of stearoyl urea; or thiocarbamyl radicals, as when using N-myristyl thiourea; or a substituted carbamyl radical

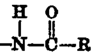


$$\left(-\overset{O}{\overset{\|}{C}}-N\begin{matrix}H\\ R^4\end{matrix}\right)$$

where R^4 is an alkyl or aryl radical, as when N-methylol N'-stearoyl-N'-ethyl urea and N'-lauroyl N-ethyl urea are employed. X may also be an aryl or alkaryl radical as when using stearanilide, a stearotoluidide, a stearoxylidide and N-benzyl stearamide and similar compounds with alkylated methylol melamine in my finishing composition.

It is not necessary that R be an aliphatic radical of at least 7 carbon atoms as in the above examples, if the long chain alkyl hydrophobic group is provided elsewhere in the compound. For example, in compounds such as N-acetyltetradecyl amine R is a small alkyl group and X is a long chain alkyl radical. Other acid amides of this type may, of course, be employed such as N-acetyloctadecyl amine, N-acetyldodecyl amine, N-acetyl 2-ethyl-hexylamine and others of similar character.

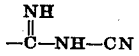
The urethanes are also examples of nitrogen-containing compounds falling within the purview of my invention and have been employed by me in conjunction with alkylated methylol melamines with very satisfactory results. These compounds are esters of carbamic acid and have the general formula



$$H-\overset{H}{\overset{|}{N}}-\overset{O}{\overset{\|}{C}}-R$$

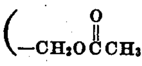
in which R is an alkoxy radical containing at least 7 carbon atoms. Examples of urethanes which may be employed are octyl carbamate, decyl carbamate, dodecyl carbamate, tetradecyl carbamate, hexyldecyl carbamate, octadecyl carbamate, etc. Substituted urethanes such as N-methyloloctadecyl carbamate, N-methyloctadecyl carbamate, N-methylol dodecylcarbamate and N-ethanol octadecyl carbamate have also been used with very good results.

A great many compounds of other types may also be employed with alkylated methylol melamines in my textile finishing composition and among these may be mentioned lauroyl dicyandiamide and stearoyl dicyandiamide in which case X in the general formula is a cyanoguanyl radical



$$-\overset{NH}{\overset{\|}{C}}-NH-CN$$

Compounds such as N-methylstearamide acetate in which X is a methoxy acetyl radical



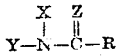
$$\left(-CH_2O\overset{O}{\overset{\|}{C}}CH_3\right)$$

may also be employed. When using alkyl esters of nitrocarbamic acid such as octyl nitrocarbamate, decyl nitrocarbamate, dodecyl nitrocarbamate, octadecyl nitrocarbamate, etc., X in the general formula is a nitro radical and R is an alkoxy radical.

Mono and diacyl substituted ammelines and melamines such as dioctanoyl ammeline, dilauroyl ammeline, monostearoyl ammeline, monolauroyl melamine, etc. may also be employed with alkylated methylol melamine. Compounds of this class have the general formula set forth above when X represents the heterocyclic triazine radical. In the case of N-cyclohexyl stearamide X is a cycloaliphatic radical.

Although most of the various compounds named above may be regarded as amides of monocarboxylic acids, my invention is by no means limited thereto since compounds may also be used such as di(octyl)oxamide, di(octyl)succinamide, di(dodecyl)succinamide, di(tetradecyl)succinamide, di(octadecyl)succinamide, di(octadecyl)adipamide, di(octadecyl)pimelamide, di(octadecyl)suberamide, and di(octadecyl)sebacamide, etc. in which both substituents may be attached to the same nitrogen atom or each nitrogen atom may contain one substituent. Other suitable compounds which may be regarded as being derived from polycarboxylic acids include succinamic acid esters such as the dodecyl esters of succinamic acid, the octadecyl ester of succinamic acid, the octadecyl ester of adipamic acid, etc. Amides of cycloalkyl, aryl and aralkyl carboxylic acids such as cyclohexyl acetic acid, benzoic acid and phenyl acetic acid which contain a long chain hydrophobe alkyl radical may also be employed in my finishing composition. Other types of compounds falling within the purview of my invention which may be used with alkylated methylol melamine to produce textile finishing compositions include for example, the N-acylated and N-carbalkoxylated urethanes such as N-lauroyl ethyl urethane, and N-carbethoxylaurylurethane, ethyl N-lauroylcarbamate and lauryl N-carbethoxycarbamate.

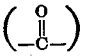
As will be obvious from a consideration of the various types of compounds named above each has the general formula



$$\mathrm{Y-\overset{X}{\overset{|}{N}}-\overset{Z}{\overset{\|}{C}}-R}$$

in which Y is hydrogen or a methylol radical, Z is oxygen or sulfur, X may be a non-functional radical such as hydrogen, alkyl, alkylol, cyano, alkylenenitrile, carbamyl, substituted carbamyl, thiocarbamyl, aryl, cyanoguanyl, alkoxy-acetyl, nitro, heterocyclic, cycloaliphatic, carbalkoxy, etc., and R may be an alkyl, alkoxy, cycloalkyl, aryl or aralkyl radical.

A great many compounds other than those falling within the general formula described above, may also be employed since, as earlier stated, my invention includes those compounds which have an alkyl radical of at least 7 carbon atoms and containing a nitrogen atom having attached thereto an acidic group and a reactive constituent selected from the group consisting of H and lower alkylol radicals. Such compounds other than those specifically described above are, for example, methyl N-tetradecyl methylsulfonamide, N-tetradecyl phenylsulfonamide, p-lauroyl phenylsulfonamide, N-p-lauramidophenylsulfonamide and the like. In these compounds, acidic groups other than the carbonyl



$$\left(\mathrm{-\overset{O}{\overset{\|}{C}}-}\right)$$

group are effective in activating the hydrogen or methylol radical attached to the amido nitrogen.

Although it has been indicated above that at least one mole of the amido compound will react with one mole of alkylated methylol melamine to produce resins of hydrophobic character, I have found that it is not necessary to use such a high proportion of the higher aliphatic amido compound to obtain satisfactory water-proofing of textile fabrics. In general I have found that the water-resistant characteristics of the treated fabrics do not improve appreciably when more than 5 parts of the amido compound are used with each 10 parts by weight of the alkylated methylol melamine. Ordinarily I employ about 1.5 parts by weight of the higher aliphatic amido compound for each 10 parts by weight of the methylated methylol melamine in my finishing compositions. Smaller proportions of this amido compound may be used with, however, a decrease in the water resistance of the treated fabric. One-half part by weight of the amido compound for each 10 parts by weight of the alkylated methylol melamine will form a finishing composition capable of rendering most textile fabrics fairly water-resistant. All the advantages of shrink resistance, crease-resistance, improved hand, etc., obtained by methylated methylol melamine alone, of course, are imparted to the fabric as a result of my treatment whether the proportion of higher aliphatic amido compound be high or low in the finishing composition.

The alkylated methylol melamine-higher aliphatic amido compound composition which I employ in my process is ordinarily, and preferably, applied to the fabrics in the form of an aqueous dispersion containing from about 2–15% or more of the above materials. These dispersions may be prepared by simply stirring a suitable amount of the amido compound dissolved in a solvent such as ethyl alcohol, isopropyl alcohol, alkyl ether of ethylene glycol, etc. into an aqueous dispersion of the alkylated methylol melamine, both solutions being preferably at temperatures of 80° F. to 140° F. When the material to be dispersed is normally a liquid it is desirable to cool it to avoid coalescence of the emulsion. Dispersing agents such as sodium isopropyl naphthalene sulfonate, dioctyl sodium sulfosuccinate, N-octadecyl disodium sulfosuccinaminate, ammonium caseinate, gelatin, glue, gum arabic, etc. may be added to either component if desired. The preparation of these dispersions is illustrated in greater detail hereinafter.

The dispersions may be applied to the fabrics in various ways known to those in the art; thus, for example, the dried fabrics to be treated may be immersed in the resin dispersion and then passed through suitable rolls as in a padder or mangle to secure uniform impregnation and to remove excess resin. The fabric, however, may be impregnated by other methods such as by spraying or with suitable boxes located on the mangle. My invention is not limited to any particular method of impregnating the fabrics and other methods will occur to those skilled in the art.

The amount of my alkylated methylol melamine-higher aliphatic amido finishing composition which may be applied to fabrics may vary considerably depending upon the nature of the fabric treated and the particular finish desired. Fabrics of wool, silk, rayon, cotton, etc. may be rendered water-repellent and substantially resistant to shrinkage by the application of from about 2 to 8% by weight of the resin-forming constituents based on the dry weight of the fabric. Smaller amounts down to 0.5% may be used with less effective results. Larger amounts as for example 8–14% by weight of the fabric will increase the water repellency of the fabric and also impart thereto a high degree of crease-resistance. With particular types of fabrics and to secure particular results it may be desirable to use larger amounts of my finishing composition, up to 30% by weight of the alkylated methylol melamine-higher aliphatic amido compound mixture as based on its weight in the finishing composition. After the fabric has been impregnated it is dried and the fabric heated at elevated temperatures to polymerize the resin.

In order to speed up the curing of the resin and decrease the heating time a suitable catalyst may be added to the aqueous finishing composition. Oxalic acid, diammonium hydrogen phosphate and methyl acid pyrophosphate are particularly good for this purpose. Other catalysts such as triethanolamine phthalate, acetic acid, mineral acids such as sulfurous acid and others may also be used. Other catalysts for the curing of alkylated methylol melamine resins are known to chemists in the resin art and may be employed.

The curing temperatures are in general quite low and may vary considerably from about 200° F. to about 300° F. with a corresponding reduction in time of cure with increase of temperature. The drying and curing operation is flexible and may be varied to suit the equipment available to the processor. High temperatures of the order of 280° F. to 300° F. will cure the resin to a suitably water-insoluble state in three or four minutes. Where facilities are not available for curing the resin at moderate temperatures within the range of about 240–280° F. the fabric after being impregnated with the desired amount of my finishing composition may be framed to width on a pin tenter, dried, batched up on a shell and allowed to stand hot to obtain a total drying and heating time sufficient to dry the cloth and insolubilize the resin in the fabric. With some fabrics a drying and curing time of two or three hours may be required at 200° F. Drying and curing times will also depend to some extent upon the effectiveness of the particular accelerator employed and upon the nature of the fabric.

After the fabric has been treated as described it should, particularly in the case of woolen goods, be given a short mild soaping which renders it soft and pliable. The fabric may then be given other usual finishing treatments such as decatizing, brushing, shearing, pressing, etc. My process may be employed with both colored and uncolored goods without appreciably affecting the color or shade and without damage to the material.

My invention will now be illustrated in detail by the following example in which the preparation of methylated methylol melamine, dispersion methods, spray drying tests, etc. are described and the quantitative results obtained are given. It should be understood, however, that although these examples may describe some of the more specific details of the invention they are given primarily for illustrative purposes and the invention in its broader aspects is not limited thereto.

960 parts by weight of formalin (37% by weight CH_2O) and 252 parts by weight of melamine were charged into a reaction vessel fitted with an agitator, thermometer, and reflux condenser and arranged so that it could be placed under a vacuum and heated. The contents of the reaction vessel were heated in one-half hour to a temperature of 62° C. and the pH adjusted to 7.5 with 2 normal NaOH. After heating for another one-half hour, a vacuum of 28 inches of mercury was applied and the mixture concentrated by removal of water. After 40 minutes heating at approximately 54° C. the heat and vacuum were removed and 1000 parts by weight of methyl alcohol containing enough 2 normal H_3PO_4 to neutralize the 2 normal NaOH added previously. The mixture at a pH of 6.4 was then heated to distill a mixture of methanol and water, anhydrous methanol being continuously added to replace the distillate. After heating in this way for about 6 to 7 hours, one part of the distillate would tolerate 100 parts of toluol and the resin was considered dried. It was then concentrated under a vacuum of 28 inches of mercury to a solids content of approximately 80%.

25 parts of the above-described 80% methylated methylol melamine, 20 parts of a 5% solution of sodium isopropyl naphthalene sulfonate and 135 parts of water were mixed and heated to approximately 110° F. Three parts by weight of methylol stearamide was dissolved in 60 parts by weight of ethyl alcohol and the solution at a temperature of about 110° F. was poured in the methylated methylol melamine solution while stirring. A stable dispersion of fine particle size was obtained. To this dispersion was then added diammonium hydrogen phosphate in the amount of 4 percent based on the total resin solids.

Samples of 80 x 80 cotton percale were impregnated with this dispersion by the dip and nip processes, regulating the take up so that the fabric contains approximately 11.5 percent by weight of the methylated methylol melamine-methylol stearamide constituents. The impregnated fabric was then dried and cured for 7 minutes at 290° F. The treated cloth samples were then given a short soaping for one-half hour at 160° F. in ½ percent soap solution, rinsed and dried. Other samples of the treated samples were soaped at 160° F. in a ½ percent soap solution for periods of 4 hours and 6 hours in order to test the permanency of the water repellent finish. Still other samples of the treated fabric were cleaned in Stoddard solvent for two hour and four hour periods at room temperature to determine the stability of the finish to dry cleaning. The samples were then subjected to a standardized water repellency test as follows:

A weighed 6 inch square of the cloth treated with the finishing composition was suspended 10 inches from a horizontal spray nozzle supplied with water at 80° F. under a hydraulic head of exactly 6 feet. The cloth was sprayed for one minute during which time 1.5 gallons of water were discharged through the nozzle. The cloth samples were then drained for 10 seconds, rolled between pieces of absorbent paper to remove surface water, and then weighed. The increase in weight was expressed as percent water absorbed, based on the original dry weight of the cloth. An untreated sample of the cloth absorbed from 90–100% of its weight of water. The results of these spray tests are in the following table.

A large number of representative long chain alkyl substituted amido and sulfonamide compounds were dispersed with methylated methylol melamine and applied to fabrics as described above. While the absolute results varied somewhat, it was found in each case that the fabric was water repellent to a very satisfactory degree. It was also found that other methods of dispersing the finishing compositions could be employed and in general the smaller the particle size of the dispersion the greater was the waterproofing effect obtained.

Methylol dodecyl carbamate was dispersed with methylated methylol melamine by dissolving the methylol dodecyl carbamate in five times its weight of ethyl alcohol and dispersing it by pouring the solution into a dispersion of methylated methylol melamine as described above. Dodecyl carbamate was dispersed with methylated methylol melamine in a similar way and applied to fabrics. Methylol stearoyl urea is a very insoluble material and more difficult to disperse. In the case of this compound it was necessary to use 70 parts by weight of Cellosolve to dissolve 3 parts by weight of methylol stearoyl urea when heated at a temperature of 180° F. The results obtained with dispersions prepared with this compound and others are also shown in the following table:

TABLE

Spray test at 80° C.

Moisture, absorption in per cent

	Soaped at 160° F. in ½% soap			Dry cleaned in Stoddard solvent (Varsol) at room temp.	
	30 min.	4 hr.	6 hr.	2 hr.	4 hr.
1.5 parts methylol stearamide + 10 parts m. m. m.[1]	27	29	28	28	37
1.5 parts methylol dodecyl carbamate + 10 parts m. m. m.[1]	27	29	28	26	33
1.5 parts dodecyl carbamate + 10 parts m. m. m.[1]	27	28	------	[2] 29	------
1.5 parts methylol stearoyl urea + 10 parts m. m. m.[1]	33	46	47	36	39
1.5 parts N-acetyl tetradecylamine + 10 parts m. m. m.[1]	28	27	------	[2] 28	------
1.5 parts stearoyl cyanamide + 10 parts m. m. m.[1]	24	28	------	[2] 24	------

[1] M. m. m. + methylated methylol melamine.

[2] 30 minutes.

The results of these spray tests show a very effective waterproofing of the samples as a result of my treatment and further show a high degree of permanency of the finish to both ordinary washing and dry cleaning processes. It was also found that the cloth also showed a very low degree of shrinking as a result of the washing.

Dispersible textile treating compositions suitable for use in my process may also be prepared by methods involving the use of less solvent and fewer manipulative steps. For example, 50 parts by weight of crude stearamide, 90 parts by weight of 95% ethyl alcohol and 17 parts by weight of a 10% solution of sodium dioctyl sulfosuccinate were placed in a reaction vessel fitted with stirrer, reflux condenser and thermometer. This mixture was heated to reflux to dissolve all of the stearamide and then it was cooled to 70° C. 6.2 parts by weight of 30% sodium hydroxide dissolved in 23.2 parts by weight of 37% formaldehyde was then added to the reaction mixture and the contents of the vessel kept at 70–75° C. for one hour. A mixture of 414 parts by weight of 80% methylated methylol melamine in methanol, 17 parts by weight of a 10% solution of sodium dioctyl sulfosuccinate and 34 parts by weight of water was added to the hot reaction mixture in the flask. This procedure gave a paste-like mass which was thoroughly stirred to insure complete mixing. The paste was then run through a homogenizer to obtain an even consistency. This product is particularly useful in the practice of my invention in that it can be readily stored and shipped and need only be diluted with water and catalyst added to obtain a dispersion of fine particle size suitable for the impregnation of fabrics in accordance with my invention.

A similar product was prepared as follows: 50 parts by weight of crude stearamide and 90 parts by weight of denatured alcohol were placed in a reaction vessel and heated to about 80° C. to obtain a clear solution. After cooling the solution 414 parts by weight of 80% methylated methylol melamine in methanol was added. 20 parts by weight of a 37% formaldehyde solution and 5.2 parts by weight of 37% sodium hydroxide were immediately added to the reaction mixture. The temperature was then slowly raised to 75° C. at which point a clear solution was obtained. Heating was continued at 70–75° C. for 1¼ hours. At the end of this time the temperature was raised to 78° C. to clear up the solution and it was then poured into a vessel and cooled slowly.

Aqueous dispersions suitable for the impregnation of fabrics were prepared by mixing 72 parts of the above described paste with 72 parts by weight of water and 20 parts by weight of a 10% solution of sodium isopropyl naphthalene sulfonate. The mixture was heated to boiling and the clear solution then further diluted with 226 parts by weight of water while rapidly stirring. To this dispersion was added 20 parts by weight of a 10% solution of diammonium hydrogen phosphate. Pieces of 80 x 80 cotton percale were then impregnated with the mixture and after curing and scouring the test pieces were found to have a water-repellency of the order shown by the results in the above table.

Carboctadecoxy cyanamide, octadecyl carbamate, methylol octadecyl carbamate, stearoylcyanamide, carboctadecoxycyanamide, diacetyldecanoguanamine, N-myristoyl thiourea, lauroylcyanamide, stearoylurea, methylol lauramide, were also dispersed with methylated methylol melamine, applied to cotton fabric and the fabric dried and heat cured as described above. These fabrics after soaping at 160° F. in ½ percent soap solution for 30 minutes showed water repellency of the order of compounds set forth in the above table. The water repellency of the fabrics was also found to be maintained despite soaping for periods of 4 hours and dry cleaning and soaping for 30 minutes.

What I claim is:

1. A textile finishing composition comprising a dispersion of an alkylated methylol melamine having alkyl groups of not more than four carbon atoms and a compound having an alkyl radical of at least seven carbon atoms and containing a nitrogen atom having attached thereto a carbonyl radical and a reactive substituent selected from the group consisting of H and alkylol radicals.

2. A textile finishing composition comprising an aqueous dispersion of methylated methylol melamine and methylol stearamide, said methylol stearamide being present in the dispersion in amounts corresponding to about 0.5 parts by weight to 5.0 parts by weight for each ten parts by weight of methylated methylol melamine.

3. A textile finishing composition comprising an aqueous dispersion of methylated methylol melamine and methylol lauramide, said methylol lauramide being present in the dispersion in amounts corresponding to about 0.5 parts to 5.0 parts by weight for each ten parts by weight of methylated methylol melamine.

4. A method of finishing a textile fabric which comprises treating the textile fabric with a dispersion of an alkylated methylol melamine having an alkyl group of not more than four carbon atoms and a compound having an alkyl radical of at least seven carbon atoms and containing a nitrogen atom having attached thereto a carbonyl radical and a reactive substituent selected from the group consisting of H and alkylol radicals, and thereafter heating the treated textile fabric to insolubilize the resin-forming constituents therein.

5. A method of finishing textile fabrics which comprises impregnating the textile fabrics with an aqueous dispersion of a methylated methylol melamine and methylol stearamide, said methylol stearamide being present in the dispersion in amounts corresponding to about 0.5 to 5.0 parts by weight for each ten parts by weight of methylated methylol melamine and thereafter heating the textile fabric within the range of 200 to 300° F. to cure the resin-forming constituents to a water-insoluble state.

6. A textile fabric of improved water repellency containing a substantially water-insoluble resinous composition comprising a heat cured mixture of an alkylated methylol melamine having alkyl groups of not more than four carbon atoms and a compound having an alkyl radical of at least seven carbon atoms and containing a nitrogen atom having attached thereto a carbonyl radical and a reactive substituent selected from the group consisting of H and alkylol radicals.

7. A textile fabric of improved water repellency containing a substantially water insoluble resinous composition comprising a heat cured mixture of a methylated methylol melamine and stearamide.

8. A textile finishing composition comprising a dispersion of an alkylated methylol melamine

having alkyl groups of not more than four carbon atoms and a compound having an alkyl radical of at least seven carbon atoms and containing a nitrogen atom having attached thereto a carbonyl radical and a reactive hydrogen substituent.

9. A textile finishing composition comprising a dispersion of an alkylated methylol melamine having alkyl groups of not more than four carbon atoms and a compound having an alkyl radical of at least seven carbon atoms and containing a nitrogen atom having attached thereto a carbonyl radical and a reactive alkylol radical.

10. A textile finishing composition comprising an aqueous dispersion of a methylated methylol melamine and an amide of an aliphatic mono-carboxylic acid of at least eight carbon atoms, said amide having a reactive hydrogen attached to the nitrogen atom thereof.

11. A textile finishing composition comprising an aqueous dispersion of a methylated methylol melamine and an amide of an aliphatic mono-carboxylic acid of at least eight carbon atoms, said amide having a reactive alkylol radical attached to the nitrogen atom thereof.

12. A textile finishing composition comprising an aqueous dispersion of an alkylated methylol melamine having alkyl groups of not more than four carbon atoms and up to a substantially equi-molecular proportion of an amide of an aliphatic mono-carboxylic acid of at least eight carbon atoms, said amide having a reaction hydrogen attached to the nitrogen atom thereof.

13. A textile finishing composition comprising an aqueous dispersion of methylated methylol melamine and up to a substantially equi-molecular proportion of stearamide.

14. A method of finishing textile fabrics which comprises impregnating the textile fabric with an aqueous dispersion of a methylated methylol melamine and an amide of an aliphatic mono-carboxylic acid of at least eight carbon atoms, said amide having a reactive hydrogen attached to the nitrogen atom thereof and being present in the dispersion in amounts up to about an equi-molecular proportion based on the content of methylated methylol melamine therein and thereafter heating the textile fabric to cure the resin-forming constituents to a water insoluble state.

15. A textile fabric of improved water repellency containing a substantially water insoluble resinous composition comprising a heat cured mixture of a methylated methylol melamine and a compound having an alkyl radical of at least seven carbon atoms and containing a nitrogen atom having attached thereto a carbonyl radical and a reactive hydrogen substituent.

16. A textile fabric of improved water repellency containing a substantially water insoluble resinous composition comprising a heat cured mixture of a methylated methylol melamine and a compound having an alkyl radical of at least seven carbon atoms and containing a nitrogen atom having attached thereto a carbonyl radical and a reactive alkylol radical.

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