This invention relates to novel textile sizing compositions, to a method of sizing textile fibers and yarns, and fabrics and to the thus sized materials.

According to this invention, textile fibers, threads or yarns are sized with a sizing composition substantially free from fats, waxes and oils and comprising a water soluble, solid, film forming polymer and at least 7%, calculated on the weight of the polymer, of a solid aliphatic carboxylic acid as defined hereinafter to produce a sized yarn or thread at least two layers thick, the inner layer consisting primarily of the polymer and the outer layer consisting primarily of the acid.

It is an object of this invention to provide novel textile sizing compositions, textile fibers, yarns and threads sized with the novel sizing composition and a method of sizing said yarns, threads and fibers with said composition.

Another object is the provision of novel sizing compositions, which are removable and dispersible in mild alkaline scour.

Still another object is the provision of novel sizing compositions comprising water soluble polymers which provide at least one of improved weaving efficiency in humid atmospheres; which are clear, self-lubricating; which shed less during weaving and substantially eliminate gummy deposits on the shuttle and loom parts; which are resistant to grease staining of the woven fabric; which reduce the number of defects and frays in the woven fabric; and which lay the fuzz on spun yarns and threads.

Other objects will be apparent to those skilled in the art to which this invention pertains.

Starch in its various forms has long been recognized as the most universal warp size. It fulfills the need for increased strength in the warp, warp that is least cut for cotton. However, starch has many deficiencies which have caused the industry to look for other sizing compositions, particularly with the advent of the many synthetic fibers. Notable among these deficiencies are shedding on the loom parts, the expense of an enzyme scour, opacity which reduces warp and pattern inspection time consuming and expensive, poor stretch, stiffness of the warp, high friction and brittleness and, with many synthetic fibers, little or no adhesion. Some of these deficiencies are diminished by compounding the starch with, e.g., penetrating, softeners and lubricants in the forms of waxes, fats and oils or other ingredients.

Unfortunately, the use of these additives creates new problems which must be solved. The net result is that starch is a poor sizing material for some materials and unacceptable for others.

Thus, although starch in one or more of its forms remains the most common size ingredient for cotton, many synthetic size materials have been developed for wool, synthetic fibers and blends thereof with cotton.

Most of these synthetic size materials are water soluble, thus overcoming the time consuming and expensive enzyme treatment necessary with starch. More recently, water soluble polyurethanes polymers from polyesters and other polyols and disocyanates have been developed which have outstanding adhesive and cohesive strength.

However, because these synthetic sizes are water soluble, they are also sensitive to humid atmospheres, becoming tacky, losing strength and are unsatisfactory or useless at relative humidities as low as 60%. As many mills are incapable of maintaining such low humidities or do not desire to do so because of the high cost in so doing, the use of these size materials has been fairly limited compared with starch, which functions well at relatively high humidities.

Moreover, many of the above-mentioned synthetic size materials have poor polymers in lubricant properties. This is overcome to some extent by the use of lubricating agents such as oils, fats, waxes, silicones, etc. However, the resulting build up on the shuttle and loom parts and the difficulty of removal of these lubricating ingredients from the fabric produces new problems that complicates the weaving or knitting step and subsequent scour.

Thus, the industry has been faced with the choice of the use of the natural products, e.g., starches, gums and fats, with their limitations and defects, or turning to the water soluble polymers which require expensive air conditioning equipment to produce the low humidities necessary with their use.

It has now been found that the properties and versatility of these water soluble polymers can be improved by incorporating them into a sizing composition as hereinafter defined. Moisture sensitivity is diminished, lubricity is highly satisfactory without producing objectionable deposits on the shuttle and loom parts. Co-loom loneliness and cohesive strength of the polymer is retained, thus providing strong yarns and threads for weaving or knitting, fuzz is laid on spun yarn, loom efficiency is improved, the clarity of clear polymers is not materially affected, thus facilitating weave and pattern inspection and the tying in of broken ends, securing is simple and inexpensive, and the sizing composition can be prepared from inexpensive ingredients.

The sizing compositions of this invention are substantially wax, fat and oil free. Substantially means less than 1%, calculated on the polymer solids, preferably less than 0.5% and more preferably free from those water insoluble materials. Whereas with starch and similar sizing materials, waxes, fats and oils are commonly employed and enhance the properties of the size, these ingredients are undesirable in the sizing compositions of this invention because of the problem of their removal, and they are unnecessary and do not contribute advantageous properties to the sizing composition. Wax, by definition, is an hydrocarbon or an insoluble ester, ordinarily of a fatty acid and a high molecular weight alcohol. Fats are greasy or oily substances similar to waxes but of a lower melting point, usually glycerides of fatty acids. Oils are liquid compounds similar to fats and waxes. Obviously, the acids employed in the sizing compositions of this invention are not within the definition of waxes, fats or oils, the latter compounds being essentially neutral compounds in their pure form.

The polymers which can be used in the sizing compositions of this invention are the water soluble, film forming polymers. As is well known in the art, it is preferred if such polymers have high cohesive strength as a film to minimize shedding during weaving or knitting. Ordinarily, preferred are the substantially neutral sizes although the acido polymers are desirable for wool and nylon. Examples of these water soluble polymers are sodium carboxymethylcellulose and other water soluble derivatives of cellulose, polyeinyl alcohol, polyeinyl acrylate, acrylamide and ammonium salts of polyeinyl acrylate, polyeinylacrylic acid, hydrolyzed styrene maleic anhydride copolymers and other copolymers of unsaturated substances containing a non-conjugated olefinic linkage and an ethylene $\alpha, \beta$-dicarboxylic acid or anhydride and mixtures of the above. Preferred are the water soluble polyurethanes, the preparation of which is disclosed in detail hereinafter. These polyurethanes perform in an outstanding fashion in the sizing compositions of this invention. Of these, those prepared from polyethylene ether...
glycols, especially those having a molecular weight of from about 4,000 to about 10,000, and aryl disoianoylates, especially tolyene-2,4-disoianoylate, are particularly desirable with those prepared in the presence of amounts of water discussed hereinafter performing in a particularly good manner.

The acids which are employed in the process of this invention are aliphatic carboxylic acids having lubricating properties. These characteristics are typified by the straight chain C12 to C19 fatty acids, e.g., lauric, myristic, palmitic, margaric and steaíc acids, and anodic-carboxylic acids, e.g., azelaic and pimelic and hydroxyalyl derivatives thereof, e.g., 12-hydroxystearic acid. In practice, these acids are often employed as a mixture, e.g., stearic and palmitic acids, sometimes with substantial amounts of unsaturated acids, e.g., oleic acid. The only requirement is that the mixture meet the requirements herein defined.

The high molecular weight fatty acids having lubricating properties, as is well known in the art. Stearic acid is an excellent example of a lubricating acid, as are the other acids in the C12 to C19 series, preferably the even numbered acids. When overcoat on a film of the water soluble polymers, employed in this invention, the acids behave in much the same manner as excellent parting agents. It is this property which enables the sizing compositions of this invention to function at high relative humidities, e.g., 65 to 85%, for the reasons discussed hereinafter. Because of their lubricating properties, they also eliminate the necessity of waxes, fats and oils which are undesirable from the standpoint of scouring and loom cleanliness.

The acids employed in the sizing compositions of this invention must be solids at room temperature and more preferably melt above 40°C. However, they must melt below 110°C to produce the continuous film characteristic of the sized yarns of this invention. It is also advantageous if the acid or mixture comprising the acid melts below 85°C so that the slashing mixture is completely liquid, thus facilitating even application on the yarn and the preparation of stable dispersions of the acid in the sizing mixture.

These acids must also be insoluble in the selected polymer and in water. Because of the highly hydrophobic properties of the aliphatic acids and the hydrophilic properties of the polymers employed herein, this limitation is readily achieved. However, these properties are vital to maintain film strength in the polymer and produce the desirable layer film characteristic of the sizing compositions of this invention. Similarly, these acids also readily disperse in water as their alkali metal salts, thus providing a self-scouring size which leaves the scoured yarn immaculately clean with little scouring effort. Because the scouring material is present on the yarn when woven, any grease spots which commonly occur during weaving are more readily removed.

The sizing compositions of this invention are characterized by producing a two layer film on the sized yarn. When the yarn is passed through an aqueous dispersion of the sizing composition and allowed to dry, the acid, by phase separation, migrates to the surface, leaving a tough film of the polymer below to bind the yarn fibers, substantially unweakened by the presence of the acid. When the yarn is heated, during or after drying, the acid melts and on cooling forms a solid, dry but lubricating the outer surface on the yarn which prevents the hydrophilic polymers from developing a tacky surface upon storage or during weaving.

To achieve this continuous outer film, it is necessary that the acid be present in an amount of at least 7%, calculated on the polymer solids, more preferably 10% or more. Lesser amounts do not produce the desired result. The exact amount of acid necessary depends in part upon the type of fiber, yarn or thread being sized and the pickup of the sizing composition thereon during sizing. Generally speaking, a pickup of at least 0.25% and preferably at least 0.50%, of the acid, calculated on the weight of the dry yarn, is necessary. Thus, if a low pickup of total solids is desired, e.g., 0.5 to 5% in the case of filament yarn, the sizing composition should contain about 13 to 50% or more acid, calculated on the polymer, whereas with spun yarns, where a 3 to 10% pickup of solids is desired, only about 7 to 30% of the acid is necessary, calculated on the polymer. The exact amount of acid necessary for optimum results depends on the selected acid and polymer, as well as other variables known to those of skill in the art.

For uniform distribution of the acid and polymer on the yarn, it will be apparent that the acid should be uniformly dispersed throughout the sizing bath. With some polymers and acids no additional dispersant is necessary because of the dispersing properties of the polymers themselves, particularly in the case of the polyelectrolytes. However, it is usually advantageous to employ substantial amounts of a surface active agent, e.g., metal salts of sulfonated and carboxylated hydrocarbons. For an excellent discussion of compounds within this general class see "Encyclopedia of Chemical Technology," 2nd ed., Interscience Publishers, Inc. (1945). Examples of these compounds are the soaps, i.e., salts of fatty acids, e.g., sodium stearate, sodium palmiate, the alkylenesulfonates, e.g., sodium dodecylbenzenesulfonate, salts of straight chain hydrocarbon sulfates, e.g., sodium lauryl sulfate. Others are the esters of alkali metal sulfoisocyanic acids, metal salts of unsaturated fatty acid esters of thiocynic acid (Igepon A) and of N-methylurea (Igepon T), and alkali metal salts of sulfonated monoglycerides. Other dispersants include the non-ionic and the cationics, which ordinarily are not preferred because of their tendency to plasticize and thus weaken some of the polymers. A compilation of commercial available surface active agents can be found in Soap and Chemical Specialties, December 1957, January, February, March and April 1958. Preferred among the surface active agents are the water-dispersible soaps, e.g., alkali metal stearate, alkali metal palmiate, etc.

As in many instances no dispersant for the acid is necessary because of the dispersing properties of the selected polymer, it will be apparent that the amount of dispersant which can be employed can be varied over a wide range when used in a particular sizing composition. Although as much as 25% or more, calculated on the polymer, can be employed, especially when the dispersant is water insoluble and has lubricating properties itself, as in the case of the alkali metal soaps, ordinarily between about 1% and 10%, calculated on the polymer is employed. It is sometimes desirable to employ an agent, in addition to the selected polymer and acid, which increases the viscosity of the slashing mixture, thereby increasing the concentration of the polymer solids on the surface of the yarn and reducing penetration thereof into the center of the yarn. This is advantageous for spun yarn where the laying down of the fuzz is an important problem. Sodium carboxymethylcellulose is excellent for this purpose as well as other water soluble derivatives of cellulose and starch. However, these ingredients merely enhance the properties of the sizing composition for specific yarns and, in fact, are neither necessary nor desirable when sizing filament yarns where a high degree of penetration is important.

Wetting agents can also be used to assist the wetting of the fibers with the sizing composition. Butanol is excellent for this purpose and can be used in substantial percentages, calculated on the polymer. It is also useful as a solvent for the acid in preparing the original dispersion. Moreover, when soap or other dispersant is employed, it tends to reduce foaming and also acts as a stabilizer for concentrated aqueous mixtures of the sizing compositions of this invention.
The sizing compositions of this invention are useful on a wide variety of textile fibers, yarns and fibers. Primarily, they are intended for warp sizing although single end sizing can also be performed with these sizes. The sizing compositions comprising polyacrylic acid or other acidic polymers are most useful on wool and nylon. The polyacrylic acid is useful on wool, and the polyacrylamide is useful on nylon. Suitable compounds include, e.g., polyelectrolytes, and especially those that are water soluble and that are able to complex with oppositely charged molecules such as proteins or DNA.

In sizing fibers, yarns or threads with the novel sizing compositions of this invention, the usual sizing techniques can be employed. The sizing solution can be maintained at room temperature or heated up to as high as the boiling point of the mixture. Usually it is desirable to heat the sizing mixture, e.g., to about 60°C to 85°C, in order to permit increased swelling.

It is necessary, to achieve the novel two phase coating on the sized material with the outer face comprising a continuous film of the selected acid, that the sizing composition be heated at some time during the sizing operation at a temperature above the melting point of the selected acid, e.g., after the sized material has passed through the sizing solution. This is most conveniently achieved by the use of heated dry cans in the conventional manner. As the yarn or thread is dried, the acid migrates to the surface, thus interfering with the yarn or fiber polymer cohesion necessary to achieve increased strength. Because the acid is heated above its melting point, as it cools, it forms a continuous outer coating by phase separation over the polymer coated fibers, thereby providing an effective non-tacky barrier for the moisture sensitive polymers. Because the acids employed in the compositions of this invention have lubricating properties, the outer film of the acid acts as an effective lubricant, thus eliminating the need for the undesirable waxes, fats and oils employed for this purpose in the sizing compositions of the prior art.

The water soluble polyalkylene ether glycol disiocyanate polyurethanes which can be employed in the sizes of this invention are prepared by reacting a substantially anhydrous polyalkylene ether glycol, e.g., having a molecular weight of from about 750 to 20,000 with about 1.5 to 1.8 molar equivalents of disiocyanate, preferably an aryl disiocyanate. The preferred molar ratio of disiocyanate to glycol is about 1.21. The term "polyalkylene ether glycol" refers to water soluble polyether glycols which are derived from alkylene oxides or glycols, e.g., those represented by the formula HO(CH₂)mOCH₂n-H, in which n is an integer from 2 to 8 and m is an integer from about 15 to about 450. Not all the alkylene radicals present need be the same, and polyether glycols containing a mixture of alkylene radicals can be used. The molecular weights of the polyalkylene ether glycols which are most useful in the sizes of this invention are from about 2,000 to 10,000 and most desirably from about 4,000 to 8,000, e.g., 5,500 to 7,000. The term includes copolymers with propylene oxide, polymethylmethylecyleglycol, polytetramethylene, and polyvinylene ether glycols. The preferred glycols are polyethylene ether glycols.

The term "substantially anhydrous" is used to define a glycol containing less than about 0.5% moisture. Although the starting polyalkylene ether glycol should be substantially anhydrous, the reaction mixture preferably is not as highly anhydrous as the reaction, to proceed in a desirable fashion, sometimes requires the presence of a trace of moisture, e.g., 100-500 parts per million calculated on the polyalkylene ether glycol, to initiate the reaction. If the polyalkylene ether glycol is dried by distilling an aromatic solvent therefrom, water is preferably added, after the addition of the disiocyanate and catalyst, in an amount of from about 50 to 500 ppm. A wide variety of disiocyanate can be used but aryl, especially monophenyl disiocyanates, are preferred. Suitable compounds include 2,4-tolylene disiocyanate, 2,6-tolylene disiocyanate, m-phenylene disiocyanate, 2,4'-dinitrodiphenylene-4,4'-disiocyanate, cyclohexyphenyl-4,4'-disiocyanate, hexamethylene disiocyanate, diphenyl-4,4'-disiocyanate, diphenylmethane-4,4'-disiocyanate, para-xylylene-4,4'-disiocyanate, naphthalene-1,4-disiocyanate and the corresponding 1,5, and 2,7 isomers thereof; fluoro-xyloxy-4,4'-disiocyanate, isophenylene-2,4-disiocyanate and diphenoxyl methylene-4,4'-disiocyanate.

Any catalyst known to be useful in the reaction of polyalkylene ether glycols with disiocyanate may be used. It has been found that particularly good results are obtained by using organo-metallie salts, e.g., cobalt naphthenate and similar salts of lead, zinc, tin, copper and manganese. Ordinarily, only a very small amount of the organo-metallie catalyst is required, e.g., from about 0.1 to 0.001% of the reactants.

The reaction is ordinarily conducted in an inert solvent to avoid working with too viscous mixtures. Generally speaking, it is preferred to operate with reaction mixtures having a viscosity of less than 1,000,000 cps and more desirably below about 800,000 cps. However, if too much solvent is employed, it tends to interfere with the reaction and slow it down. This effect can, to a certain extent, be overcome by the use of large amounts of catalyst. It is ordinarily desirable to employ only that amount of solvent which will impart a viscosity to the reaction mixture in the range of about 100,000 to 1,000,000 cps, preferably around 300,000 to 800,000 cps. With toluene at 75 to 85°C, employing a polyalkylene ether glycol of a molecular weight in the range of 5,500 to 7,000, this can be accomplished at an initial concentration of about 80% solids. As the reaction proceeds, the increasing molecular weight of the reaction product increases as the viscosity of the reaction mixture, thus necessitating the gradual addition of more solvent throughout the reaction if about the same viscosity is to be maintained, e.g., until a final concentration of as low as 50% solids is reached. This serves two purposes, i.e., maintaining the desired viscosity and also slowing down the reaction. Thus, as the reaction product approaches water insolubility or gelation because of its increasing molecular weight, the reaction rate tends to slow down due to the presence of the increasing amounts of solvent, thereby providing more leeway in the time at which the chain termination agent should be added to prevent the production of a water insoluble reaction product. The amount of solvent employed can be varied considerably, e.g., from about 10% to 60% of the total reaction mixture.

The temperature of the polymerization reaction can be varied over a considerable range so long as the reaction is stopped at the desired point. The reaction proceeds slowly unless the temperature is above about 65°C. However, the temperature should not exceed 150°C, and preferably should not exceed 110°C. The preferred range is from about 70°C to 95°C. The reaction time is a function of such factors as temperature, mixing speed, ratio of reactants, water concentration and amount of catalyst and solvent employed.

Oxidation and discoloration of the reaction product can be avoided by conducting the polymerization reaction in an inert atmosphere, e.g., nitrogen, which also aids in the production of a more uniform reaction product.

When the desired viscosity is reached, the resulting polymer can be chain terminated in the manner described hereinafter, or epoxide modified as described below and then chain terminated.

Epoxide modified polyalkylene ether disiocyanate polyurethanes are prepared by the reaction of a polyalkylene ether glycol disiocyanate polyurethane described above with an epoxide. Tillion, a 30% solution of the above described polymer in methylene chloride, can be reacted with 10% glycidyl ether of polyethylene glycol of a molecular weight in the range of 5,500 to 7,000. The reaction is conducted at 60°C in the presence of a catalytic amount of dibutyltin oxide. The reaction mixture is then adjusted to a pH of from 8.5 to 9.5 and poured into a large quantity of water and then expressed into a further quantity of water until the final product is at a pH of from 6.5 to 7.0. It is then acidified to a pH of from 3.0 to 3.5 and finally adjusted to a solid content of from about 45% to 55%. The resulting product is then compressed into tablets of a desirable size and shape and dried at a temperature of from about 90°C to 100°C and a pressure of from about 0.5 to 1.0 psig to a solid content of from about 90% to 95%.
ferred procedure involves adding the epoxide to the reaction mixture for a few minutes, e.g., 1 to 15 minutes, before the polymer is chain terminated.

Examples of epoxides, preferably the compounds which can be prepared from α-glycols, are the lower hydrocarbon, i.e., containing from 2 to 12 carbon atoms, epoxides including styrene oxide, α-phenyl propylene oxide, trimethylene oxide and the other lower alkylene oxides, i.e., epoxides containing from 2 to 8, preferably 2 to 4, carbon atoms, inclusive, e.g., ethylene oxide, propylene oxide, butylene oxide, isobutylene oxide.

The amount of epoxide which can be added to the polyols or polyether glycol diisocyanate polymer can be varied over a wide range, i.e., from about 0.1 mole per mole of diisocyanate to the theoretical 2 moles per mole of diisocyanate or more. Conveniently, and preferably if the epoxide is volatile, an excess of the epoxide can be added and the excess removed by distillation or evaporation after the reaction has proceeded to the desired extent. The epoxide modified portion of the polymerization reaction is ordinarily conducted in substantially the same manner as the preceding portion of the polymerization reaction.

The chain termination of a polymer is a well known reaction in polymer chemistry. In this step, the terminal reactive groups of the polymer are reacted with a non-chain extending compound which inactivates these groups. In the instant polymer, the reactive terminal groups are isocyanate groups. These groups merely require a non-chain extending compound having an active hydrogen, i.e., those hydrogen atoms which display activity according to the well known Zerewitinoff test. See J. Am. Chem. Soc., 49, 3181 (1927). For a discussion of diisocyanate chemistry, see National Aniline Division of Allied Chemical and Dye Corporation Technical Bulletin 1–17 and the references cited therein. The preferred chain terminating agents are those having only one active hydrogen. Suitable chain terminating agents are alcohols, water, ammonia, primary amines, cyclic secondary amines, acids, inorganic salts having an active hydrogen, mercaptans, amides, alkyl amine, oximes, etc. Lower alkanols, i.e., containing from one to eight carbon atoms, inclusive, are preferred, especially those containing less than 4 carbon atoms.

The minimum amount of chain terminating agent which should be employed will vary according to the ratio of diisocyanate to hydroxy groups present in the reaction mixture. It is preferred to add at least several molar equivalents, calculated on the diisocyanate used, as a safety factor.

The total polymerization time can vary considerably depending, in part, on the molecular weight of the starting polyalkylene ether glycol, the reaction temperature, the catalyst and amount of solvent employed. The extent limits of reaction time, under a particular set of reaction conditions, can be determined by removing sampling from the reaction mixture from time to time, chain terminating the sample with a lower alkanol, e.g., ethanol, and then making a 25% aqueous solution thereof, while removing whatever reaction solvent may be present, and determining the viscosity at 25°C. of at least 2,000 cps., and preferably at least 8,000 or more, the desired reaction product can be obtained from the reaction mixture upon chain termination thereof.

Another convenient index for determining the course of reaction is the viscosity of the reaction mixture. If the reaction is conducted at 75 to 85°C. with toluene as a reaction solvent, a 65% solution of the reaction mixture should have a viscosity in the range of 50,000 to 1,000,000 cps. As stated before, such a reaction mixture produces a highly satisfactory size if chain terminated at a viscosity of around 200,000–800,000 cps.

Improved polymers are obtained if a water soluble polyurethane described above, usually as an aqueous 3−7 lution, is reacted with an aldehyde, thereby producing a polymer having improved properties, including increased film strength and increased resistance to heat and atmospheric moisture.

A wide variety of aldehydes can be employed, both aromatic and aliphatic. The aldehydes can be monomelic, polyaldehyde. It is preferred that the aldehyde has no groups other than aldehyde which can be reacted with the starting polymer. Examples of aldehydes, e.g., aliphatic preferably containing 1 to 12 carbon atoms, include formaldehyde, acetaldehyde, propionaldehyde, butyraldehyde, n-butyraldehyde, formylglycolic acid, n-butyldihydroxyethylene, and other lower aliphatic and alcyclic monofunctional aldehydes, glyoxal, pyruvaldehyde, ethylglyoxal, amylyglyxal, and other α-carbonyl lower aliphatic aldehydes, benzaldehyde, cinnamaldehyde, phenylacetaldelyde, α-naphthaldehyde, pyrocatechualdehyde, veratraldehyde, α-formylphenone, α-formylfuraron, and other substituted and unsubstituted aromatic aldehydes, dialdehyde starch and other aldehyde carbohydrates and aldehydic cellulose materials. The preferred aldehydes are the lower, i.e., containing from 1 to 12 carbon atoms, inclusive, aliphatic and carbocyclic aromatic monomalealdehydes. Formaldehyde is the aldehyde of choice.

The aldehyde reaction can be conducted at any convenient temperature, e.g., 0°C. to 100°C., although a temperature between about 20°C. and 85°C. is more desirable and between about room temperature and about 70°C. preferred. The reaction can be conducted at any pH between 3 and about 10. Outside this range, the starting and resulting polymer tend to be unstable. Ordinarily, it is preferred to stay within the range of about 3.5 to about 9. If it is intended to produce an acid size, e.g., one particularly useful on wool or nylon, the reaction can be conducted at a pH between 3 and about 10. Outside the range, the resulting polymer tends to be unstable. For example, the propylene oxide modified polymer produced from polyethylene ether glycol 6,000 and tolylene 2,4-diisocyanate, as a 25% aqueous solution with a viscosity of about 15,000 cps., can react, at room temperature, with 0.05%, calculated on polymer solids, of formaldehyde at a pH of 4 and 0.1% formaldehyde at a pH of 5 without producing a water insoluble gel in several days, whereas at a pH of 8, the same polymer can react with 1−3% or more formaldehyde and still produce water soluble reaction products. It will be apparent from the above that, when operating at an alkaline pH, care should be taken that the pH does not drift during the reaction to the acid side. This can be avoided by conducting the reaction in the absence of oxygen to prevent the air oxidation of the aldehyde or buffering the solution, e.g., with Na₂HPO₄.

Also, when operating on the acid side, if the reaction product is approaching water insolubility, the pH of the reaction mixture can be adjusted by adding an organic or inorganic base, e.g., ethyl amine, sodium hydroxide or ammonia, to render the reaction product less reactive toward the residual aldehyde.

In any case, the reaction is conducted for a time less than that required to produce a water insoluble reaction product. The preferred starting reaction mixture is preferably aqueous, e.g., with 20−30% polymer solids. These mixtures can have a viscosity from about 2,000 to as
high as 50,000 cps. or higher at 25° C. With a highly reactive aldehyde, e.g., formaldehyde, on the acid side at, e.g., 90° C., the desired viscosity can be reached in a matter of minutes. At room temperature, the viscosity can slowly rise for several weeks or more on the acid side employing 0.05% of the aldehyde or when employing a slowly reacting aldehyde such as daldichydro starch. On the alkaline side, any viscosity change usually is less rapid.

The stability of all of the above-described polyurethane as aqueous solutions is excellent so that they may be stored in any of the usual liquid or solid forms. Their good water solubility permits mixing at any point in the mill thereby eliminating a separate cooking operation, e.g., as required for starch and other conventional sizes.

The following preparations and examples are illustrative of the process and products of this invention, but are not to be construed as limiting.

**PREPARATION I**

Dissolve 11,250 g. of polyethylene ether glycol having an average molecular weight of 6,000 (range of 5,500 to 7,000) in 3000 cc. of hot toluene. Evacuate the reactor several times, breaking the vacuum each time with dry nitrogen. Distill the mixture under nitrogen at atmospheric pressure until about 2,600 cc. of toluene has been removed, to dry the solution. The polyethylene glycol which in all respects appears to be dry, is indicated to contain about 0.4% weight of water according to the Karl Fischer test at this time. Mix 13.2 g. of a 6% solution of cobalt naphthenate in xylene with an amount of dry toluene equivalent to the amount removed from the reactor and add the solution to the reaction. After 10 minutes, add 393 g. of toluene-2,4-diisocyanate in 750 cc. of dry toluene. After 4 minutes, add 50 drops (2.5 cc.) of water over a one-minute period at 82° C. Flush the reactor with more nitrogen and continue the reaction at about 93° C. for about 2 to 2.5 hours, until the viscosity reaches about 500,000 cps. Then slowly add 3,000 cc. of dry toluene over a 30 to 45 minute period, to keep the viscosity of the mixture at about 500,000 cps., followed by 375 cc. of propylene oxide in 750 cc. of dry toluene. After 10 to 15 minutes, remove about 350 cc. of distillate at reduced pressure and then add 375 cc. of ethyl alcohol. After 10 to 15 minutes, add about 16 liters of cold water and mix thoroughly. Slowly distill the toluene at reduced pressure until about 45 to 60° C. until a clear solution is obtained. This solution, diluted to 25% solids with water, has a viscosity of at least 8,000 cps. and ordinarily is at least 12,000 cps.

To 113.5 kilograms of a 25% solution of the above-described polyurethane add sufficient dilute sodium hydroxide at room temperature to bring the pH of the solution to about 8.5. Evacuate the reactor several times, flushing each time with nitrogen. Heat the mixture under nitrogen to about 65-70° C. and add 2.5% aqueous formaldehyde, calculated on the polymer solids (690 cc. of 37% formaldehyde). Heat under nitrogen for one hour at 70° C. Store at room temperature for about a week.

**PREPARATION II**

Heat under nitrogen with rapid stirring, 3,750 grams of polyethylene ether glycol having an average molecular weight of about 6,000 in a 1.5-liter 3-neck round bottom flask having a reflux condenser and 250 ml. of toluene and then stirring the solution at reduced pressure. Add 4.4 grams of a solution of 6% cobalt naphthenate in dry xylene to 1,250 ml. of dry toluene and then add the resulting solution slowly to the melt at 75-80° C. with stirring. Add 131 grams of tolylene 2,4-diisocyanate over a 10 minute period and stir another 10 minutes. There should be a rise at this stage. After this point, add dropwise about 10 to 20 drops (0.4 to 0.8 gram) of water. Continue stirring at 80-95° C. for 15 minutes and then reduce the stirring speed. When the viscosity reaches about 200,000 cps. at about 85° C. (90 to 120 minutes), slowly add 1,250 ml. of dry toluene (90 to 120 minutes) without lowering the temperature below 80° C. or markedly reducing the viscosity. After the toluene addition has been completed and the viscosity reaches about 300,000 cps. (about 15 minutes), equip the flask with a reflux condenser and then slowly add 104 grams of propylene oxide. After about 10 minutes, remove the excess propylene oxide by distilling at reduced pressure. When the viscosity reaches 500,000 cps. (usually 5 to 15 minutes) terminate the reaction by stirring in 100 grams of absolute ethanol. Stir in 5 liters of hot water and stop the heating. Transfer the reaction mixture to a 20 liter flask, add 7.5 liters of water and distill off the toluene at reduced pressure. There is obtained a clear, amber solution of about 25% solids having a viscosity of about 10,000 to 25,000 cps. at 25° C.

An equivalent amount of polyethylene ether glycol having a molecular weight of about 4,000 can be substituted for the PEG 6000.

Follow the procedure of Preparation II, but eliminate the propylene oxide step. A polymer is produced having properties similar to the polymer produced in Preparation II, but is somewhat less resistant to atmospheric moisture.

**PREPARATION III**

The procedure of Preparation II is followed exactly except that 120 g. of tolylene 2,4-diisocyanate and 3.2 g. of the 6% cobalt naphthenate solution is employed. The viscosity at 25° C. of a 25% aqueous solution of a polymer produced according to this procedure is about 10,000 cps.

Similar results are obtained if the procedure of Preparation III is followed except that methanol, isopropanol or n-butanol is substituted for the ethanol. The polymer thus produced is substantially identical to that obtained according to the procedure of Preparation II. 187 g. of diphenyl methane 4,4'-diisocyanate can be substituted for the tolylene 2,4-diisocyanate.

**PREPARATION IV**

Prepare a 25% aqueous solution of the polymer produced according to Preparation II. Adjust the pH to 8.2 with 1 N sodium hydroxide. Under a blanket of nitrogen and with stirring, add sufficient 10% formalin to give 1.25% formaldehyde, calculated on the polymer solids. Heat the mixture to 70° C. for 30 minutes and then cool. The mixture is poured into a blanket of ice. Similar results are obtained employing starting polymers having about the same viscosity and prepared in the manner described in Preparation II employing a polyethylene ether glycol having a molecular weight of about 4,000 or polypropylene ether glycol having a molecular weight of about 600.

2.25% formaldehyde can be substituted for the 1.25% formaldehyde and, if desired, heating of the formaldehyde mixture can be omitted.

**PREPARATION V**

Adjust the pH of a 25% aqueous polyurethane solution having a viscosity of about 10,000 cps. at 25° C., prepared according to the procedure of Preparation III, to 4.0 with H3PO4. Add 0.05%, calculated on the polymer solids, of formaldehyde as a 1% aqueous solution. Maintain the mixture for 10-20 days at about 28° C. in the substantial absence of oxygen. The resulting mixture has a viscosity of about 130,000 to 180,000 at 28° C. as a 25% aqueous solution.

The procedure of Preparation V can be followed, except that the mixture can be aged for two days at 30° C. rather than 10-20 days at 28° C. Similarly, acetaldheyde can be substituted for the formaldehyde.

**PREPARATION VI**

Follow the procedure of Preparation V, but employ
7% benzaldehyde instead of formaldehyde, calculated on the polymer solids. The resulting mixture will contain sufficient excess benzaldehyde to produce a water insoluble polymer if heated for any appreciable time. The product should therefore not be heated to any significant extent prior or during use.

If only 1% benzaldehyde, calculated on the polymer solids is employed, the resulting mixture can be heated, e.g., at 50°C until the desired viscosity is reached in the reaction product. If only 0.1% benzaldehyde is employed, the reaction can be conducted at 70°C.

Similarly, an equal molar amount of 30% aqueous glyoxal can be substituted for the formaldehyde. The reaction rate is somewhat slower than with formaldehyde.

**PREPARATION VII**

To a 25% aqueous solution of a polyurethane prepared according to the procedure of Preparation II, add 10%, by weight of polymer solids, of a dialkylstarch prepared by oxidizing starch with HIO₄ ("Simstar-S," Miles Chemical Co.). Cure at room temperature for one or more days. Upon prolonged ageing, care should be taken, e.g., by the exclusion of oxygen or oxidizing agents and/or by the addition of alkaline or buffering agents, to prevent the pH of the resulting mixture from drifting to the acid side. If an acid water soluble product is desired, the amount of starch to be added should be substantially reduced.

**Example I**

A sizing composition is prepared as follows: 70 lbs. of a 30% aqueous solution of the polyurethane produced according to the procedure of Preparation I is mixed at about 60°C with 10.5 lbs. of a 10% aqueous suspension of high titer soap (made from Armour Neo-Fat 18 stearic acid and sodium carbonate), 6.3 lbs. of high titer stearic acid, 4.2 lbs. of a 1.5% aqueous suspension of carboxymethylcellulose (Hercules high viscosity), 42 lbs. of n-butyl alcohol and 217 lbs. of water. The resulting mixture is diluted with water to 6% polymer solids (8.3% total solids) to give a sizing mixture having a viscosity of about 400 c.p.s. at 70°C. A warp of spun 65% Dacron polyester 35% cotton 24/1 singles is passed into the sizing mixture at a box temperature of 65°C and then passed through squeeze rolls at a pressure adjusted to provide a pickup of about 3.7% total solids, calculated on the weight of the dry fabric. The warp is then passed over dry cans, the first three of which are heated at 100°C, the next two at 105°C, the next two at 110°C, and the last at 115°C. The split bars are placed as compactly and as closely as possible to the dry cans.

The sized warp has a considerable increase in yarn strength and no objectionable tackiness is observed at 75% relative humidity. The yarns have a coating of a film of the polyurethane which film has an outer coating of stearic acid. Whereas a warp sized only with the polyurethane becomes a tacky upon standing for several days at 75% relative humidity and produces a buildup on the shuttle and a sticky deposit on the reeds, heddles and drop wires, the above warp remains tack-free on standing and does not produce a significant amount of deposit on the shuttle or loom parts during weaving. The incident of defects in the woven fabric is considerably less than the same fabric sized with conventional sizes. The sized warp is clear, permitting ready identification of the pattern and individual ends and scours size-free in a mild alkaline scour.

Excellent weaving results are obtained when a warp of spun 65% Dacron polyester 35% rayon 24/1 singles is substituted for the polyester-cotton warp employed in Example I and is slashed and woven in the manner described above.

The weight of polyurethane solution can be varied from about 50 to 100 lbs., the stearic acid varied from 3 to 10 lbs. and the sodium carboxymethylcellulose eliminated, if desired, in the above composition.

Similar results are obtained if palmitic acid, a mixture of palmitic and stearic acid, myristic or lauric acid is substituted for the stearic acid.

**Example II**

A sizing composition is prepared as follows: 550 lbs. of a 30% aqueous solution of the polyurethane, produced according to the procedure of Preparation I, is mixed at about 65°C with 50.7 lbs. of a hot mixture consisting of a 10% aqueous suspension of high titer soap (sodium stearate), 24.8 lbs. of high titer 12-hydroxy-stearic acid, 33 lbs. of n-butyl alcohol and 2,301.5 lbs. of water to make up 3,000 lbs. The resulting mixture is diluted with water to 5% polyurethane solids (6.67% total solids).

A warp of 40/27/14 filament Dacron polyester is passed into the sizing mixture at a box temperature of about 65°C and then passed through soft squeeze rolls at high pressure. The warp is then dried over hot dry cans. The solids pickup will vary from about 1.8 to 2.2%, calculated on the weight of the dry warp.

The sized warp is clear, slick, tack-free at 65% relative humidity. The filaments have a coating of a film of the polyurethane, which film has an outer coating of 12-hydroxy-stearic acid. No build-up of size on the shuttle or loom parts occurs during weaving. Fabric woven from the warp has a low incidence of defects and has a finished appearance even before scouring. The size is readily removed with a mild alkaline scour.

**Example III**

Follow the procedure of Example II, but substitute an equal weight of pine oil for the 12-hydroxy-stearic acid. 40/27/14 filament Dacron polyester warp sized with this sizing composition weaves smoothly without significant build-up on the shuttle or loom parts. Loom efficiency is considerably higher than when employing conventional sizes, e.g., sodium polycrylate, stearic or polyethylene emulsions.

Similarly, azelaic acid can be substituted for the pine oilic acid with comparable results.

**Example IV**

Prepare a dispersion of 28.1 g. of a 26.7% aqueous solution of a polyurethane having a viscosity of about 13,000 c.p.s., prepared according to the procedure of Preparation II, 2.25 g. of stearic acid (Armour Neo-Fat 18), 1.5 g. of butanol and 118.2 g. of water. Pass a spun 65% Dacron polyester 35% cotton warp through this dispersion, containing about 5% polymer solids, at room temperature and through squeeze rolls set to provide about a 5% pickup, based upon polymer solids. Pass the wet warp over heated dry cans and then, while hot, over split rods. The resulting warp has increased strength, the fuzz is laid and weaving efficiency is high with minimal shuttle pickup or shedding at 70% relative humidity.

Similar results are obtained with an equal weight of a polyurethane prepared according to the procedure of Preparation VII and having a viscosity, as a 30.1% solution, of about 80,000 c.p.s., is substituted for the polyurethane employed in Example IV.

**Example V**

Follow the procedure of Example IV, but substitute an equal weight of 12-hydroxy-stearic acid for the stearic acid. The resulting dispersion is a solid which is excellent for laying the fuzz of spun yarn, e.g., single end cotton or Dacron polyester or a warp thereof, by passing the yarn into the dispersion and allowing the warp to pass therethrough to lay the fuzz as the yarn is passed therethrough. The yarn can then be air dried with hot air. The pickup of the size on the yarn can be controlled by the squeeze rolls or by varying the concentration of solids therein.

Similar results are obtained when an equal weight of a
polyurethane, prepared according to the procedure of Preparation VII, and having a viscosity as a 30.1% solution of about 80,000,000, and being substituted for the polyurethane employed in Example V.

**Example VI**

Prepare a dispersion of 15 g. of polyvinyl alcohol (Elvanol 50–42), 5% sodium stearate, 15% stearic acid, and 20% of butanol, calculated on the polyvinyl alcohol, in 129 g. of water. The resulting dispersion, containing 10% polyvinyl alcohol makes an excellent size for 15 denier monofilament nylon. Compared with straight polyvinyl alcohol, moisture sensitivity is reduced and lubricity is considerably improved. Mild alkaline scour readily removes the size.

Follow the procedure of Example VI, but substitute an equal weight of sodium polyacrylate for the polyvinyl alcohol. An excellent sizing composition for nylon warps is thus obtained.

What is claimed is:

1. An aqueous dispersion of a textile sizing composition, substantially free of fats, waxes and oils and consisting essentially of (a) a water soluble, film forming polymer which is a solid at room temperature and thin films of which tend to become tacky and lose cohesive strength in humid air, whereas having a relative humidity above 60%, and (b) at least 7%, calculated on the weight of the polymer, of an aliphatic carboxylic acid having lubricating properties, which is insoluble in said polymer and in water, which is a solid at room temperature and melts below 110°C, and which, as an alkali metal salt, disperses in water, said dispersion producing a two layer film, with the acid as the outer layer, when cast and dried.

2. A dispersion according to claim 1 wherein the acid is a monobasic acid, containing from 12 to 22 carbon atoms, inclusive, the carboxyl radical of which is attached to a long straight chain saturated aliphatic group.

3. A dispersion according to claim 2 wherein the acid is selected from the group consisting of saturated straight chain fatty acids and hydroxylated derivatives thereof.

4. A dispersion according to claim 3 wherein the acid melts below 100°C, and is present in an amount of at least 10%, calculated on the weight of the polymer.

5. A dispersion according to claim 4 wherein the acid comprises stearic acid.

6. A dispersion according to claim 4 wherein the acid comprises 12-hydroxystearic acid.

7. An aqueous dispersion of a textile sizing composition consisting essentially of (a) a water soluble, film forming, polyisocyanate ether glycol polyurethane which is a solid at room temperature and has a viscosity, as a 25% aqueous solution, of at least 2,000 cps. at room temperature, and (b) at least 7%, calculated on the weight of the polyurethane, of an aliphatic carboxylic acid having lubricating properties, which is insoluble in said polymer and in water, which is a solid at room temperature and melts below 110°C, and which, as an alkali metal salt, disperses in water, said dispersion producing a two layer film, with the acid as the outer layer, when cast and dried.

8. A dispersion according to claim 7 wherein the acid is selected from the group consisting of saturated straight chain fatty acids containing from 12 to 22 carbon atoms, inclusive, and hydroxylated derivatives thereof.

9. An aqueous dispersion of a textile sizing composition consisting essentially of (a) a water soluble, film forming, polyisocyanate ether glycol polyurethane which is a solid at room temperature and has a viscosity, as a 25% aqueous solution, of at least 6,000 cps. at room temperature, and (b) at least 7% by weight, calculated on the weight of the polyurethane solids, of a saturated straight chain monobasic aliphatic carboxylic acid containing from 12 to 22 carbon atoms, inclusive, which melts between 40° and 110°C, said dispersion having a pH between about 7 and about 10 at a 5% concentration and producing a two layer film, with the acid as the outer layer, when cast and dried.

10. A dispersion according to claim 9 wherein the acid comprises stearic acid.

11. A dispersion according to claim 10 wherein the acid comprises 12-hydroxy stearic acid.

12. An aqueous dispersion of a textile sizing composition substantially free of fats, waxes and oils and consisting essentially of (a) a water soluble, film forming, polyethylene ether glycol polyurethane which is a solid at room temperature and has a viscosity, as a 25% aqueous solution, of at least 10,000 cps. at room temperature, and (b) at least 10% by weight, calculated on said polyurethane, of stearic acid, said dispersion having a pH between about 7 and about 10 at a 5% concentration and producing a two layer film, with the acid as an outer layer, when cast and dried.

13. An aqueous dispersion of a textile sizing composition substantially free of fats, waxes and oils and consisting essentially of (a) a water soluble, film forming, polyethylene ether glycol polyurethane which is a solid at room temperature and has a viscosity, as a 25% aqueous solution, of at least 10,000 cps. at room temperature, and (b) at least 10% by weight, calculated on said polyurethane, of 12-hydroxystearic acid, said dispersion having a pH between about 7 and 10 at a 5% concentration and producing a two layer film, with the acid as an outer layer, when cast and dried.

14. An aqueous dispersion of a textile sizing composition substantially free of fats, waxes and oils and consisting essentially of (a) a water soluble, film forming, aldehyde modified, polyethylene ether glycol aryl polyurethane wherein the polyethylene ether polymeric units have a molecular weight between about 4,000 and 8,000 and said polyurethane is a solid at room temperature and has a viscosity as a 25% aqueous solution, of at least 10,000 cps. at room temperature, and (b) at least 10%, calculated on the weight of the polyurethane, of saturated fatty acid containing from 12 to 22 carbon atoms, inclusive, and (c) a surface active compound selected from the group consisting of metal salts of sulfated, sulfonated, and carboxylated hydrocarbons, said dispersion having a pH between about 7 and 9 at a 5% concentration and producing a two layer film, with the acid as the outer layer, when cast and dried.

15. A dispersion according to claim 14 wherein the surface active compound is an alkali metal soap.

16. A dispersion according to claim 15 wherein the polyurethane is a polyethylene ether glycol toluene 2,4-diisocyanate polyurethane, the acid comprises stearic acid present in an amount between about 10 and about 50%, calculated on the weight of said polyurethane, and the surface active compound comprises sodium stearate, present in an amount between about 1 and 25%, calculated on said polyurethane.

17. An aqueous dispersion of a textile sizing composition substantially free of fats, waxes and oils and consisting essentially of (a) a water soluble, film forming, aldehyde modified, polyethylene ether glycol aryl polyurethane wherein the polyethylene ether polymeric units have a molecular weight between about 4,000 and 8,000 and said polyurethane is a solid at room temperature and has a viscosity as a 25% aqueous solution of at least 10,000 cps. at room temperature, and (b) between about 10% and 50%, calculated on the weight of said polyurethane, of 12-hydroxystearic acid, and (c) a surface active agent selected from the group consisting of metal salts of sulfated, sulfonated and carboxylated hydrocarbons, said dispersion having a pH between about 7 and 9 at a 5% concentration and forming a two layer film, with the acid as the outer layer, when cast and dried.

18. A dispersion according to claim 17 wherein the surface active component comprises sodium stearate, present in an amount between about 1% and 25%, calculated on said polyurethane.
19. A sized textile yarn, said size being substantially free of fats, waxes and oils consisting essentially of an inner layer of a substantially continuous film of a water soluble polymer which is a solid at room temperature and which becomes tacky and loses cohesive strength in humid atmospheres having a relative humidity above 60%, and of a substantially continuous outer layer of at least 7%, calculated on the weight of the polymer, of an aliphatic carboxylic acid having lubricating properties, which is insoluble in said polymer and in water, which is a solid at room temperature and melts below 110° C. and which, as an alkali metal salt, disperses in water, the layers being formed by heating the yarn above the melting point of the acid after applying the polymer and acid simultaneously to the yarn as an aqueous dispersion.

20. A sized yarn according to claim 19 wherein the polymer is a polyalkylene ether glycol polyurethane and wherein the acid is a saturated straight chain monobasic aliphatic carboxylic acid containing from 12 to 22 carbon atoms, inclusive, and is present in an amount of at least 10%, calculated on the weight of the polymer, and at least 0.25%, calculated on the weight of the yarn.

21. A sized yarn according to claim 19 wherein the inner layer consists essentially of a polyalkylene ether glycol polyurethane having a viscosity, as a 25% aqueous solution, of at least 2,000 cps. at room temperature, and the outer layer comprises 12-hydroxystearic acid.

22. A sized yarn according to claim 20 wherein the inner layer consists essentially of a polyalkylene ether glycol polyurethane having a viscosity, as a 25% aqueous solution, of at least 2,000 cps. at room temperature, and the outer layer comprises stearic acid.

23. A method of sizing textile yarns comprising passing a yarn end into an aqueous, substantially fat, wax and oil free dispersion consisting essentially of (a) about 0.5% to 15.0% of a water soluble, film forming polymer which is a solid at room temperature and thin films of which tend to become tacky and lose cohesive strength in humid atmospheres, and (b) at least 7%, calculated on the polymer, of an aliphatic carboxylic acid having lubricating properties, which is insoluble in said polymer and in water, which is a solid at room temperature and melts below 110° C., and which, as an alkali metal salt, disperses in water, in a manner so as to provide a pickup of at least 0.5% of the polyurethane solids when the yarn is filament and at least 2% when the yarn is spun, the concentration of said acid in said dispersion being sufficient to provide a pickup thereof of at least a 0.25% under the selected conditions, all calculated on the weight of the dry yarn, and then drying said yarn and heating said yarn at a temperature above the melting point of said acid.

24. The method of claim 23 wherein the acid consists essentially of a monobasic acid, containing from 12 to 22 carbon atoms, inclusive, the carboxyl radical of which is attached to a long straight chain saturated aliphatic group.

25. The method of claim 24 wherein the acid comprises those selected from the group consisting of fatty acids and hydroxy fatty acids.

26. A method of sizing textile yarns comprising passing a yarn end into an aqueous, substantially fat, wax and oil free dispersion consisting essentially of (a) about 0.5% to 15.0% of a water soluble, film forming polyalkylene ether glycol polyurethane which is a solid at room temperature and thin films of which tend to become tacky and lose cohesive strength in humid atmospheres, and (b) at least 7%, calculated on the polyurethane, of stearic acid, in a manner so as to provide a pickup of at least 0.5% of the polyurethane solids when the yarn is filament and at least 2% when the yarn is spun, the concentration of said acid in said dispersion being sufficient to provide a pickup thereof of at least 0.25% under the selected conditions, all calculated on the weight of the dry yarn, and then drying said yarn and heating said yarn at a temperature above the melting point of said acid.

27. A method of sizing textile yarns comprising passing a yarn end into an aqueous, substantially fat, wax and oil free dispersion consisting essentially of (a) about 0.5% to 15.0% of a water soluble, film forming polyalkylene ether glycol polyurethane which is a solid at room temperature and thin films of which tend to become tacky and lose cohesive strength in humid atmospheres, and (b) at least 7%, calculated on the polyurethane, of 12-hydroxystearic acid, in a manner so as to provide a pickup of at least 0.5% of the polyurethane solids when the yarn is filament and at least 2% when the yarn is spun, the concentration of said acid in said dispersion being sufficient to provide a pickup thereof of at least 0.25% under the selected conditions, all calculated on the weight of the dry yarn, and then drying said yarn and heating said yarn at a temperature above the melting point of said acid.

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