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TEXTILE SIZING AGENTS PLASTICIZED WITH BLENDS OF WATER-SOLUBLE POLYHYDROXY ALCOHOLS, ALIPHATIC AMIDES AND HYDROXY ACIDS

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This invention relates, in general, to new and improved sizing compositions and in particular to those sizing compositions which are uniquely suited for use in sizing nylon yarn. More particularly, the invention relates to the novel compositions which are employed to plasticize these sizing agents.

It is common practice in the textile industry today to apply sizing agents such as, gelatin, glue, starch, casein and like materials, to various types of textile yarns which are to be woven or knitted. These sizing compositions are employed to provide the yarn with protection both during the actual weaving or knitting thereof and during those steps connected with the weaving or knitting processes. The choice of a suitable textile sizing composition is dependent upon many factors. Thus, for example, the size selected must not reduce the efficiency of the weaving or knitting processes. The size must uniformly coat the individual ends with a strong, flexible protective film. Moreover, the size must be such that it can be readily incorporated into an aqueous solution and easily applied to the yarn as such. Furthermore, the sizing agent must not adversely affect the finished handle of the fabric and it must be easily removable in conventional scouring operations. Nylon is one of the strongest fibers known in the art today. However, despite this, nylon, like most textile yarns, must also be sized with a tough flexible film to enable it to withstand mechanical stress and abrasive forces encountered during weaving or knitting. However, notwithstanding the similarity between nylon and other types of textile yarns in this respect, very many of the compositions which are eminently well suited for sizing yarns other than nylon are not at all suitable for sizing nylon yarn. The present invention is concerned chiefly with the development of a suitable nylon sizing agent.

The material most widely used for sizing nylon yarn today is polyacrylic acid. However, even though a conventional polyacrylic acid size provides the yarn with a superior adhesive coating, bonding the filament with a strong film which is readily removed by conventional scouring processes, such a size has certain undesirable characteristics. It has been found that conventional polyacrylic acid size becomes extremely brittle in a relatively short period of time. As a result, sharp-edged flaking of the size on the yarn invariably occurs causing filament breakage in the looms. The problem posed by the brittleness of a size is not a new one to the textile industry, however. It has been encountered many times before when a yarn other than nylon was to be sized with a sizing agent other than polyacrylic acid. In order to remedy this, certain materials having plasticizing properties have been incorporated into the sizing agents to impart pliability and flexibility to the protective film formed by these sizing agents. In the past, many compositions have been found to function satisfactorily as plasticizers in sizing compositions designed particularly for use on yarns other than nylon. Of these, probably the most widely

known are the sulfated oils used in conjunction with gelatine for warp sizing cellulosic fibers. However, just as the compositions employed in the art for sizing yarns other than nylon yarn were, for the most part, entirely unsuited for sizing nylon yarns, the plasticizing agents employed in these prior art sizing compositions have been found to be totally unsuited for plasticizing polyacrylic acid. The reason for this is the many and varied properties required of a plasticizing agent when used in conjunction with a particular sizing composition. In the first place, the plasticizer must be compatible with the sizing agent. It must show no tendency whatsoever to bleed or separate from the sizing agent. Thus, for example, when sizing nylon yarn with polyacrylic acid, the plasticizer employed must be compatible with polyacrylic acid. Moreover, the plasticizer must exert, uniformly and completely, its plasticizing effect throughout the sizing composition. Also, the plasticizer must impart a high degree of pliability and flexibility to the protective size film. Moreover, the size must be rendered pliable and flexible even when the proportion of plasticizer in the plasticized sizing agent is relatively small. Furthermore, in addition to these specific functions, the plasticizer composition must scour off completely, together with the sizing material, in the last scouring bath. Many plasticizing agents have been disclosed in the art as suitable for use in combination with polyacrylic acid nylon sizing compositions. However, few if any of these have thus far proven to be satisfactory in every regard.

It is an object of this invention to provide novel plasticizing compositions.

It is a further object of this invention to provide economical sizing compositions, especially nylon sizing compositions, which provide full and complete protection to yarns during all the steps in the process involved in the manufacture of fabrics therefrom.

A further object of this invention is to provide a novel plasticizing composition suitable for incorporation into conventional polyacrylic acid nylon size which when used, even in relatively small quantities, results in the production of a sizing composition for nylon yarn which will neither crack nor dust off nor chafe the nylon yarn and which will impart to the size the necessary flexibility and pliability.

A still further object of this invention is to provide a novel plasticizing composition which can be completely removed by conventional scouring procedures from the yarn to which it has been applied.

Other objects of this invention will be obvious and will in part appear hereinafter.

It has been discovered that compositions which comprise a blend of (1) water-soluble, low molecular weight, organic, hydroxy and polyhydroxy acids and certain derivatives thereof, (2) water-soluble, low molecular weight, aliphatic amides and certain derivatives thereof and (3) water-soluble, polyhydroxy alcohols are superior plasticizing compositions. The low molecular weight, hydroxy or polyhydroxy acid constituent, or the derivative thereof, comprises from about 40% to about 90% by weight of the solids in the plasticizing agents. The water-soluble, low molecular weight nitrogen-containing compound comprises from about 1% to about 50% by weight of the solids in the plasticizer. The polyhydroxy alcohol comprises from about 1% to about 30% by weight of the solids in the plasticizer.

The polyhydroxy alcohol constituent of our novel plasticizing composition can be any one of the water-soluble, low molecular weight alkylene glycols such as, for example, ethylene glycol, propylene glycol, or butylene glycol or water-soluble polymers of ethylene glycol containing up to about 15 ethylene oxide residues such

as, for example, diethylene glycol, triethylene glycol, etc. Moreover, glycerine can also be employed as the polyhydroxy constituent of our novel plasticizing compositions. Also, if desired, mixtures of two or more materials from any of the above-defined classes can instead be employed. The expression "low molecular weight" employed heretofore to describe the alkylene glycols includes those alkylene glycols having a carbon chain length of from 2 to about 8 carbon atoms. In preparing our novel compositions, the polyhydroxy alcohol comprises broadly from about 1% to about 30% by weight and preferably from about 3% to about 15% by weight of the solids in the plasticizer.

Another essential component of our plasticizing compositions is a water-soluble, low molecular weight aliphatic nitrogen-containing compound or a mixture thereof. Thus, for example, a water-soluble, low molecular weight aliphatic amide such as, formamide, acetamide, proionamide, butyramide, valeramide or urea or any other similar low molecular weight amide such as, for example, acrylamide, can be used in the production of our novel plasticizer compositions. Moreover, water-soluble derivatives of these amides in which either or both of the hydrogen atoms attached to the nitrogen atom have been replaced by an organic radical, such as a water-soluble alkyl or dialkyl derivatives of an amide, for example, dimethyl formamide, can be used in preparing the plasticizer blend of our invention. Furthermore, instead of these amides, water-soluble products produced by condensing at least one mole of a water-soluble, low molecular weight amide or a derivative thereof with at least one mole, or as many as 15 moles, of ethylene oxide can be used. Thus, for example, the product resulting from the condensation of equimolar quantities of acetamide and ethylene oxide, the product resulting from the condensation of a mole of formamide with two moles of ethylene oxide or the product resulting from the condensation of urea with one and one-half moles of ethylene oxide are well suited for use in the preparation of our novel plasticizer as are all other such condensates. The amide or the amide-ethylene oxide condensate component of our novel blend comprises, broadly, from about 1% to about 50% by weight of the solids in the plasticizer. Preferably, however, the amide or amide derivative comprises from about 3% to about 30% by weight of the solids in the plasticizing compositions.

The final ingredient used in compounding our novel plasticizing compositions is a water-soluble organic acid having a carbon chain length of from 2 to about 6 carbon atoms or mixtures thereof. The suitable acids are selected from that group of low molecular weight organic acids in which at least one hydrogen atom attached to a carbon atom in the carbon chain has been replaced by a hydroxyl group, such as for example, glycollic acid, lactic acid, gluconic acid, etc. and derivatives of said acids, such as, diglycollic acid. However, if desired instead of these acids, water-soluble condensation products thereof, such as, are produced by reacting the acid or a mixture of these acids with at least equimolar quantities of ethylene oxide or ethylene glycol and with as many as 15 moles of ethylene oxide or glycol for each mole of acid can be used in preparing our plasticizer compositions. This component of the blend comprises broadly from about 40% to about 90% by weight of the solids in the plasticizer, but preferably it comprises from about 70% to about 90% by weight of the solids in each product.

The precise manner in which the component ingredients of our novel plasticizing composition are admixed is not at all critical to the operability of the invention. Accordingly, if desired, a wholly water-free mix of the components which comprise the plasticizer can be made or, in the alternative, an aqueous solution of the blend can be prepared. When, as is done in the preferred embodiment of the invention, the plasticizing composition of the invention is produced in the form of an aqueous solution, any one of a number of procedures may be followed. Thus, if desired, a water-free blend of the materials which comprise the plasticizers can first be prepared and subsequently admixed with and dissolved in water. In the alternative, an aqueous solution of any one or more of the components can be initially obtained or prepared and subsequently admixed with the other water-free component or components or with aqueous solutions thereof. However, even though the plasticizer compositions of the invention are prepared preferably in the form of aqueous solutions, nothing in this disclosure should be construed as indicating that an anhydrous blend of the materials which comprise the plasticizing compositions cannot be produced and used as such to plasticize a textile sizing composition. It has been found, however, that by preparing the plasticizer blend in the form of an aqueous solution, the incorporation and dissolution of the plasticizer in the aqueous size solution will be greatly facilitated. The production of plasticized sizing compositions using our novel plasticizers, however, will be more fully described hereinafter. Preferably, in preparing our novel plasticizer compositions, the aliphatic amide or amide derivative and the polyhydroxy alcohol constituent of the plasticizer, both water-free, are added to and dissolved in an aqueous solution of the low molecular weight hydroxy or polyhydroxy acid. Obviously, the concentration of the aqueous acid solution at the time the amide or amide derivative and the polyhydroxy alcohol are added there to is not in the least critical since, in fact, if desired, the materials could be admixed to form the novel plasticizer in the total absence of water. However, before proceeding to the step of incorporating the plasticizer into the aqueous solution of the sizing composition, for example an aqueous polyacrylic acid solution, it is preferred that the concentration of the aqueous solution of the plasticizing composition be adjusted so that the total solids in the aqueous plasticizing composition is within the range of from about 65% to about 75% by weight. However, notwithstanding the precise sequence in which the ingredients of the plasticizer are admixed and regardless of whether an anhydrous or an aqueous solution of the plasticizer is prepared, the plasticizers of our invention will exhibit highly superior plasticizing properties when incorporated into a conventional size.

New and improved plasticized nylon sizing compositions are readily prepared by incorporating our novel plasticizers into conventional aqueous polyacrylic acid sizes. This step involves merely a simple mixing operation. Polyacrylic acid, a resin produced by the polymerization of acrylic acid usually in the presence of a catalyst, is commercially available either in the form of an aqueous solution of said resin or as a dispersion of said resin in an organic solvent. In preparing the plasticized nylon sizing compositions of the present invention, an aqueous solution of polyacrylic acid is employed. Thus, for our purposes, a commercially available aqueous polyacrylic acid solution is well suited for use. However, if available, the anhydrous resin could be used provided, of course, that prior to the use of the said resin, the anhydrous material is dissolved in water. The concentration of the aqueous polyacrylic acid solution which is used in the preparation of our novel plasticized sizing compositions can be varied within rather wide limits. For the most part, however, practical considerations will determine the permissible maximum and minimum concentration limitations. By this is meant that since the novel plasticizing agents of our invention, are to be incorporated into the aqueous polyacrylic acid solution and since the viscosity of the polyacrylic acid solution will vary directly with the concentration of the polyacrylic acid dissolved therein and since the ease with which our novel plasticizing agents are incorporated into the aqueous polyacrylic acid solution will depend to a large extent

upon the fluidity of the aqueous polyacrylic acid solution, the concentration of this solution should be such that the novel plasticizers of the invention can be readily and completely incorporated therein. Preferably, a commercially available aqueous polyacrylic acid solution which has a concentration of at least about 25% by weight of polyacrylic acid and a viscosity, as determined using a Brookfield viscosimeter, of from about 300 to about 600 centipoises is employed. A particular feature of our invention resides in the fact that, in the preparation of our novel sizing compositions, it is never necessary to employ an aqueous polyacrylic acid solution which contains less than about 25% by weight of polyacrylic acid. As a result of this, the necessity of shipping and storing cumbersome volumes of plasticized nylon sizing compositions is wholly and entirely obviated. In the nylon industry, of course, the normal procedure is to treat the yarn with a sizing agent, said sizing agent being in the form of an aqueous solution having a concentration of from about 5% to about 10% by weight of total solids. The expression "total solids" which is used in various instances throughout the specification and in the claims should be understood as meaning the weight of all of the non-aqueous constituents which comprise the aqueous solution. Our products are of such nature that the final plasticized sizing compositions can be manufactured for, sold and shipped to and stored by the ultimate consumer for extended periods of time as relatively concentrated solutions and, when and as needed in the mill, readily diluted with water to the desired concentration.

The sequence in which the aqueous solution of polyacrylic acid and the plasticizing agent are admixed in the preparation of the plasticized nylon size is of no importance whatsoever to the operability of the invention. Blending of these ingredients into a plasticized nylon size can be and is preferably accomplished, however, at temperatures at or around room temperature. If desired, temperatures higher than room temperature can be employed to facilitate complete dissolution of the plasticizing composition in the aqueous polyacrylic acid solution. Such higher temperatures, however, are not at all necessary to the complete operability of the invention.

The plasticized nylon sizing compositions of this invention are produced in the form of aqueous solutions, the total solids, i. e. non-aqueous constituents, in said solutions being broadly within the range of from about about 5% to about 50% by weight and preferably within the range of from about 20% to about 30% by weight. Polyacrylic acid comprises from about 60% to about 95% by weight of the total solids in the products and the novel plasticizing compositions of this invention comprise from about 5% to about 40% by weight of the total solids in the product. Preferably, however, polyacrylic acid comprises from about 75% to about 90% by weight and the plasticizer from about 10% to about 25% by weight of the total solids in the plasticized sizing compositions.

The novel sizing compositions of this invention can be applied to nylon yarn in the same manner in which the prior art sizing agents are customarily applied. These compositions do not necessitate the use of any special equipment nor do they require a knowledge of any special techniques which are not already entirely familiar to textile operators. Generally, the sizing compositions of this invention will be applied to nylon yarn by the conventional sizing or slashing processes employed in the art. However, since the techniques employed in the various mills do vary somewhat, this invention is not limited by the manner in which the novel plasticized sizing compositions are used. The best method for sizing nylon yarn with the compositions of this invention can be readily determined in the mill by making trial runs using the precise conditions which will be employed in the actual mill operations.

The plasticized sizing compositions of the present invention have been found to be superior in every regard to all of the other nylon sizing compositions known in the art. Thus, the plasticizing compositions of this invention are completely compatible with polyacrylic acid. On standing for extended periods of time, the plasticizer shows no tendency whatsoever to separate from the polyacrylic acid solution. As a result of the outstanding stability of these products, complete and uniform plasticity is achieved throughout the size. The plasticized sizing compositions of this invention provide coatings of protective film to the surface of nylon yarn which are both flexible and pliable. This film neither cracks nor tends to dust off nor does it chafe the nylon fabric which it envelops. A particular and an outstanding feature of the present invention is the fact that substantially greater quantities of our novel plasticized sizing compositions can be deposited on nylon yarn than can be deposited, with any beneficial results, when the sizing agent employed is conventional polyacrylic acid nylon size. This is due to the enhanced plasticity of the protective film which is formed by our products. Thus, by the use of our products nylon yarn can be afforded greater protection during weaving or knitting and cleaner woven fabrics can be ultimately obtained. Finally, the plasticized sizing compositions of our invention can be, and are, readily and completely removed from the yarns to which they have been applied by conventional scouring methods.

We have not as yet definitely established why the compositions of our invention function so successfully as plasticizing agents for polyacrylic acid nylon size. Apparently, however, since some of the ingredients employed in preparing our novel products have been used before, either alone or in combination with other materials, in certain textile-treating compositions and since no extraordinary plasticizing properties are attributed to these materials, the outstanding results that we have observed are attributable to the specific combination of ingredients and the quantity of each in our compositions.

Moreover, although the plasticizing compositions of the present invention are particularly designed for use in conjunction with polyacrylic acid nylon size and even though this use has been emphasized in the present description, the disclosure should not be construed as indicating that our novel plasticizers are suited solely for use in plasticizing polyacrylic acid. Thus, we have found that in addition to polyacrylic acid other textile sizing agents as, for example, gelatin, polyvinyl alcohol, and the like are adequately plasticized by our novel products. Furthermore, synthetic resins of the type produced by copolymerizing a mixture of monomers, at least one of the constituents of said monomeric mixture being a carboxylic acid type composition, can be plasticized by our novel products.

For a fuller understanding of the nature and objects of the invention, reference should be had to the following examples which are given merely as further illustrations of the invention and are not to be construed in a limited sense. All parts are given by weight.

Example I

In this example, 90 parts of an aqueous glycollic acid solution (70% glycollic acid by weight on a dry basis) were admixed with a mixture of 4 parts of formamide, 3 parts of diethylene glycol and 3 parts of water. This mixture was stirred until the resulting product was uniform. 13.0 parts of this homogeneous mixture were thereafter added to 363.6 parts of an aqueous solution of polyacrylic acid (25% polyacrylic acid by weight). The addition was accompanied by complete and thorough stirring. When uniformity were achieved, an additional 23.3 parts of water was added to the product following which said product was once again thoroughly stirred. The composition which resulted was a clear, slightly viscous, straw colored liquid. This straw colored liquid

will be referred to in the description which follows directly hereinafter as the plasticized polyacrylic acid size.

In order to evaluate the plasticizing effect exerted by the composition of this example a 10.0 gram sample of the aforementioned plasticized polyacrylic acid size was weighed out and poured onto a glass plate (3" x 6"). For control purposes a 10.0 gram sample of conventional unplasticized aqueous polyacrylic acid solution (25% by weight) was poured onto a similar glass plate. The glass plates were thereafter placed in an oven and heated at a temperature of from about 70° C. to about 80° C. for a period of about four hours. At the end of this time the glass plates were removed from the oven and allowed to cool to room temperature. While cooling, the thin films which had formed were pulled off each of the plates. Visual inspection of the film formed by the polyacrylic acid plasticized by the composition of this example revealed it to be clear, transparent and extremely flexible and pliable. Visual inspection of the film formed by the unplasticized polyacrylic acid revealed it also to be clear and transparent but, rather than being flexible and pliable, it was extremely brittle.

The above described procedure is an accurate and widely recognized method for evaluating plasticizers. It is an especially valuable means for observing and examining the plasticizing effect exerted by a composition on a conventional polyacrylic acid size. By proceeding in the prescribed manner the compatibility of the plasticizer with the sizing composition and the physical characteristics of the film or size formed by the plasticized sizing composition, including the smoothness, the flexibility and pliability of the film, can be observed. The results obtained in the present instance indicated that the composition of this example was highly suited for plasticizing conventional polyacrylic acid nylon size.

In view of these results an aqueous solution of polyacrylic acid containing the plasticizer composition of this example was used, at a temperature of 120° F., in the slasher pan of a Johnson warp sizer to warp size 40 denier nylon yarn. Because of the extraordinary plasticity attained by the incorporation of our plasticizer into conventional polyacrylic acid nylon size, the yarn was sized with a solution containing about 7½% by weight of solids instead of the customary 5% by weight of solids. The solution of the size was produced in the following manner. The plasticizer of this example (70% solids) was added to an aqueous solution of polyacrylic acid (25% by weight) in such proportions that the solution obtained contained about 10 parts of polyacrylic acid (dry basis) for each part of the plasticizer (dry basis). This solution was then diluted with water until the solution contained about 25% by weight of solids. The concentration of solids in this solution was further decreased by adding thereto, for each 2.5 pounds about one gallon of water. The blend was stirred thoroughly and thereafter used to size the 40 denier nylon yarn. After sizing, they arn was subjected to a conventional weaving operation. During this operation the plasticized size performed in a highly efficient manner. The size furnished complete and adequate protection to the yarn from the abrasive forces and mechanical stresses encountered during weaving. The size neither cracked nor dusted off during weaving. Furthermore, the plasticized sizing agent was readily and completely removed by conventional scouring processes and a much cleaner woven fabric was obtained.

Example II

In this example, a plasticizing composition, having the formula given below, was prepared in the same manner as was the plasticizer of Example I:

	Parts
Glycollic acid (70% soln. by weight)	41.2
Diethylene glycol	20.6
Formamide	20.6
Water	17.6

A plasticized polyacrylic acid nylon size was thereafter prepared using about 13 parts of the above described plasticized (70% by weight solids) and about 363.6 parts of an aqueous solution of polyacrylic acid (25% acid by weight). The sizing composition was prepared by simple mixing of the two ingredients. Water was added to this aqueous mixture to lower the concentration of solids therein to about 25% by weight. The composition obtained was a clear, very slightly viscous, straw colored liquid.

A 10.0 gram sample of the material was weighed out and poured onto a glass plate (3" x 6"). For control purposes a 10.0 gram sample of unplasticized aqueous polyacrylic acid solution (25% by weight) was poured onto a similar glass plate. The glass plates were thereafter placed in an oven and heated at a temperature of from about 70° C. to about 80° C. for a period of about four hours. After being thus heated, the glass plates were removed from the oven and allowed to cool to room temperature. On cooling, a thin film formed on each of the plates. Visual inspection of the film which was formed by the sample containing the plasticizer of this example revealed it to be clear, transparent and extremely flexible and pliable. Visual inspection of the film which was formed by the unplasticized aqueous polyacrylic acid sample was clear and transparent but very brittle.

Example III

In this example, a plasticizer was prepared by admixing the following materials at room temperature:

	Parts
Glycollic acid (70% solution by weight)	75.0
Diethylene glycol	10.0
A condensate produced by reacting one mole of formamide with two moles of ethylene oxide	15.0

This composition was used to plasticize polyacrylic acid in the following manner. An aqueous solution of polyacrylic acid and the composition of this example was prepared. The total solids in said solution was 25% by weight of which polyacrylic acid constituted 90% by weight and the composition of this example 10% by weight. A 10 gram sample of this material and a 10 gram sample of unplasticized aqueous polyacrylic acid (25% by weight) were applied to separate glass plates (3" x 6"). The plates were immediately thereafter placed in an oven heated at a temperature of from about 70° C. to about 80° C. for a period of about four hours. At the end of this time, the glass plates were removed from the oven and allowed to cool to room temperature. On cooling, a thin film formed on the surface of each of the plates. Visual inspection of the film which was formed by the sample containing the novel plasticizer of this example revealed it to be clear, transparent and extremely flexible. Inspection of the film which was formed by the conventional, unplasticized aqueous polyacrylic acid solution revealed that, while it was clear and transparent, it was very brittle.

Example IV

A plasticizer was prepared by admixing the following materials at room temperature:

	Parts
Glycollic acid (70% solution by weight)	75.0
Diethylene glycol	5.0
A condensate prepared by reacting one mole of urea with 1½ moles of ethylene oxide	12.5
Water	7.5

This composition was used to plasticize polyacrylic acid nylon size and its plasticizing properties were evaluated in precisely the same manner as that described in Example III. The plasticized aqueous polyacrylic acid solution produced herein formed a clear, transparent, flexible film while the unplasticized control sample of aqueous polyacrylic acid formed a clear but brittle film.

Example V

In this example a plasticizer was produced by admixing the following materials at room temperature:

	Parts
A condensate of one mole of glycollic acid with 4.2 moles of ethylene oxide	70.0
Glycerine	10.0
Formamide	20.0

This composition was used to plasticize polyacrylic acid nylon size and its plasticizing properties were evaluated in precisely the same manner as that described in Example III. The plasticized aqueous polyacrylic acid solution produced herein formed a clear, transparent, flexible film. The unplasticized polyacrylic acid formed a brittle film.

Example VI

In this example, a plasticizer was produced by admixing the following materials at room temperature.

	Parts
A condensate of one mole of diglycollic acid and 2¼ moles of ethylene oxide	60.0
Diethylene glycol	15.0
Formamide	25.0

This composition was used to plasticize polyacrylic acid nylon size and its plasticizing properties were evaluated in precisely the same manner as that described in Example III. The plasticized aqueous polyacrylic acid solution produced herein formed a clear, transparent, flexible film while the unplasticized aqueous polyacrylic acid solution which was employed for comparative purposes formed a clear, transparent but very brittle film.

Example VII

A plasticizer was prepared by admixing the following materials at room temperature

	Parts
A condensate of one mole of diglycollic acid and 2¼ moles of ethylene oxide	50
Diethylene glycol	5
A condensate of one mole of urea and 1½ moles of ethylene oxide	15
Water	30

This composition was used to plasticize polyacrylic acid nylon size and its plasticizing properties were evaluated in precisely the same manner as that described in Example III. The plasticized aqueous polyacrylic acid solution produced herein formed a clear, transparent, flexible film while the unplasticized aqueous polyacrylic acid employed for control purposes formed a clear, transparent and extremely brittle film.

Example VIII

A plasticizer composition was prepared by admixing the following material in the proportions indicated:

	Parts
Glycollic acid (70% by weight)	61
Formamide	18
A condensate of 1 mole of a polyethylene glycol (molecular weight 400) with 7.7 moles of ethylene oxide	9
Water	12

This composition was used to plasticize polyacrylic acid nylon size and its plasticizing properties were evaluated in precisely the same manner as that described in Example III. The plasticized aqueous polyacrylic acid solution produced herein formed a clear, transparent, flexible film whereas the unplasticized aqueous polyacrylic acid solution used for comparison formed a clear, transparent and brittle film.

Example IX

A plasticizer having the following formulation was prepared:

	Parts
Glycollic acid (70% by weight)	67
Ethylene glycol	12
Dimethyl formamide	11
Water	10

This composition was used to plasticize polyacrylic acid nylon size and its plasticizing properties were evaluated in precisely the same manner as that described in Example III. The plasticized aqueous polyacrylic acid solution produced herein formed a clear, transparent, flexible film while the control sample, that is the unplasticized aqueous polyacrylic acid, formed a clear, transparent and extremely brittle film.

Example X

In this example, a plasticizer was produced by mixing the following materials at room temperature:

	Parts
Lactic acid (85% by weight)	60
Diethylene glycol	15
A condensate of one mole of formamide and 2 moles of ethylene oxide	15
Water	10

This composition was used to plasticize polyacrylic acid nylon size and its plasticizing properties were evaluated in precisely the same manner as that described in Example III. The plasticized aqueous polyacrylic acid solution produced herein formed a clear, transparent, flexible film while the unplasticized aqueous polyacrylic acid solution employed for control purposes formed a clear, transparent but brittle film.

Example XI

A plasticizer was produced by admixing the following materials at room temperature:

	Parts
Glycollic acid (70% by weight)	60
A condensate of one mole of a polyethylene glycol (molecular weight 400) and about 7.7 moles of ethylene oxide	7.0
Acrylamide	21.0
Water	12.0

This composition was used to plasticize polyacrylic acid nylon size and its plasticizing properties were evaluated in precisely the same manner as that described in Example III. The plasticized aqueous polyacrylic acid solution produced herein formed a clear, transparent, flexible film while the control sample, that is the unplasticized aqueous polyacrylic acid solution, formed a clear, transparent but brittle film.

Having described our invention what we claim as new and desire to secure by Letters Patent is:

1. A sizing composition for nylon yarn consisting essentially of an aqueous solution of a (1) blend of (*a*) from about 1% to 30% by weight of a water-soluble polyhydroxy alcohol selected from the group consisting of glycerine, alkylene glycols having a carbon chain length of from 2 to 8 carbon atoms and polymers of ethylene glycol containing up to 15 moles of ethylene oxide, (*b*) from about 1% to about 50% by weight of a water-soluble, low molecular weight, aliphatic nitrogen-containing compound selected from the group consisting of aliphatic amides containing from 1 to 5 carbon atoms, mono and dialkyl derivatives thereof and derivatives of said amides which are the condensation products of said amides with up to 15 moles of ethylene oxide and (*c*) from about 40% to about 90% by weight of a compound selected from the group consisting of diglycollic acid, water-soluble, organic carboxylic acids having a carbon chain length

of from 2 to 6 carbon atoms in which at least one hydrogen atom attached to a carbon atom in the carbon chain thereof has been replaced by a hydroxyl radical and derivatives of said acids which are the condensation products of said acids with up to 15 moles of a compound selected from the group consisting of ethylene oxide and ethylene glycol, and (2) polyacrylic acid, the total solids in said solution being from about 5% to about 50% by weight, said blend furnishing from about 5% to about 40% by weight and polyacrylic acid furnishing from about 60% to about 95% by weight of said solids.

2. A sizing composition for nylon yarn consisting essentially of an aqueous solution of a (1) blend of (*a*) from about 1% to 30% by weight of a water-soluble polyhydroxy alcohol selected from the group consisting of glycerine, alkylene glycols having a carbon chain length of from 2 to 8 carbon atoms and polymers of ethylene glycol containing up to 15 moles of ethylene oxide, (*b*) from about 1 to about 50% by weight of a water-insoluble, low molecular weight, aliphatic nitrogen-containing compound selected from the group consisting of aliphatic amides containing from 1 to 5 carbon atoms, mono and dialkyl derivatives thereof and derivatives of said amides which are the condensation products of said amides with up to 15 moles of ethylene oxide and (*c*) from about 40% to about 90% by weight of a compound selected from the group consisting of diglycollic acid, water-insoluble, organic carboxylic acids having a carbon chain length of from 2 to 6 carbon atoms in which at least one hydrogen atom attached to a carbon atom in the carbon chain thereof has been replaced by a hydroxyl radical and derivatives of said acids which are the condensation products of said acids with up to 15 moles of a compound selected from the group consisting of ethylene oxide and ethylene glycol, and (2) polyacrylic acid, the total solids in said solution being from about 20% to about 30% by weight, said blend furnishing from about 10% to about 25% by weight and polyacrylic acid furnishing from about 75% to about 90% by weight of said solids.

3. The sizing composition of claim 2 in which said blend consists essentially of an aqueous solution of (*a*) a water-soluble polyhydroxy alcohol selected from the group consisting of glycerine, alkylene glycols having a carbon chain length of from 2 to 8 carbon atoms and polymers of ethylene glycol containing up to 15 moles of ethylene oxide, (*b*) a water-soluble, low molecular weight aliphatic nitrogen-containing compound selected from the group consisting of aliphatic amides containing from 1 to 5 carbon atoms, mono and dialkyl derivatives thereof and derivatives of said amides which are the condensation products of said amides with up to 15 moles of ethylene axide and (*c*) a compound selected from the group consisting of diglycollic acid, water-soluble organic carboxylic acids having a carbon chain length of from 2 to 6 carbon atoms in which at least one hydrogen atom attached to a carbon atom in the carbon chain thereof has been replaced by a hydroxyl group and derivatives of said acids which are the condensation products of said acids with up to about 15 moles of a compound selected from the group consisting of ethylene oxide and ethylene glycol, the polyhydroxy alcohol employed comprising from about 3 to about 15% by weight of the solids in said blend, the water-soluble aliphatic nitrogen containing compound employed comprising from 3% to about 30% by weight of the solids in said blend and the water-soluble acids and derivatives thereof comprising from about 70% to about 90% by weight of the solids in said blend.

4. The sizing composition of claim 2 in which said blend consists essentially of an aqueous solution of (*a*) an alkylene glycol having from 2 to about 8 carbon atoms, (*b*) a water-soluble low molecular weight aliphatic amide containing from 1 to 5 carbon atoms and (*c*) a water-soluble organic carboxylic acid having a carbon chain length of from 2 to 6 carbon atoms in which at least one hydrogen atom attached to a carbon atom in the carbon chain has been replaced by a hydroxyl radical, the alkylene glycol comprising from about 1% to about 30% by weight, the water-soluble nitrogen-containing compound comprising from about 1% to about 50% by weight and the water-soluble acids comprising from about 40% to about 90% by weight of the solids in said blend.

5. The sizing composition of claim 2 in which said blend consists essentially of an aqueous solution of (*a*) an alkylene glycol having from 2 to 8 carbon atoms, (*b*) a water-soluble, low molecular weight aliphatic amide containing from 1 to 5 carbon atoms and (*c*) a water-soluble derivative of an organic carboxylic acid having a carbon chain length of from 2 to 6 carbon atoms in which at least one hydrogen atom attached to a carbon atom in the carbon chain has been replaced by a hydroxyl radical, said derivative being the condensation product of said acid with up to 15 moles of a compound selected from the group consisting of ethylene oxide and ethylene glycol, the alkylene glycol comprising from about 1% to about 30% by weight, the water-soluble nitrogen containing compound comprising from about 1% to about 50% by weight and the water-soluble acid derivative comprising from about 40% to about 90% by weight of the solids in said blend.

6. The sizing composition of claim 2 in which said blend consists essentially of an aqueous solution of (*a*) an alkylene glycol having from 2 to about 8 carbon atoms, (*b*) a water-soluble low molecular weight aliphatic amide containing from 1 to 5 carbon atoms and (*c*) a compound selected from the group consisting of diglycollic acid, water-soluble organic carboxylic acids having a carbon chain length of from 2 to 6 carbon atoms in which at least one hydrogen atom attached to a carbon atom in the carbon chain thereof has been replaced by a hydroxyl radical and derivatives of said acids which are the condensation products of said acids with up to 15 moles of a compound selected from the group consisting of ethylene oxide and ethylene glycol, the alkylene glycol comprising from about 1% to about 30% by weight, the water-soluble nitrogen-containing compound comprising from about 1% to about 50% by weight and the water-soluble acids and derivatives comprising from about 40% to about 90% by weight of the solids in said blend.

7. The sizing composition of claim 6 in which said amide is a condensate of a water-soluble, low molecular weight aliphatic amide and up to 15 moles of ethylene oxide.

8. The sizing composition of claim 2 in which the total solids content of said blend is from about 65% to about 75% by weight of said blend, the remainder being water.

9. The sizing composition of claim 8 in which said water-soluble polyhydroxy alcohol is glycerine.

10. The sizing composition of claim 8 in which said water-soluble polyhydroxy alcohol is an alkylene glycol having a carbon chain length of from 2 to 8 carbon atoms.

11. The sizing composition of claim 8 in which said water-soluble polyhydroxy alcohol is a polymer of ethylene glycol containing up to 15 moles of ethylene oxide.

12. A sizing composition for nylon yarn consisting essentially of an aqueous solution of a (1) blend of an aqueous solution of glycollic acid, formamide and diethylene glycol, said glycollic acid comprising from about 70% to about 90% by weight, said formamide comprising from about 3% to about 30% by weight and said diethylene glycol comprising from about 3% to about 15% by weight of the total solids in said blend, the total solids content in said blend being from about 65% to about 75% by weight thereof, and (2) polyacrylic acid, the total solids in said solution being from about 20% to about 30% by weight, said blend furnishing from about 10% to about 25% by weight and polyacrylic acid furnishing from about 75% to about 90% by weight of said solids.

13. A sizing composition for nylon yarn consisting essentially of an aqueous solution of a (1) blend of an

aqueous solution of glycollic acid, diethylene glycol and a condensate of one mole of urea with one and one-half moles of ethylene oxide, said glycollic acid comprising from about 70% to about 90% by weight, said diethylene glycol comprising from about 3% to about 15% by weight and said urea-ethylene oxide condensate comprising from about 3% to about 30% by weight of the total solids in said blend, the total solids in said blend being from about 65% to about 75% by weight thereof and (2) polyacrylic acid, the total solids in said solution being from about 20% to about 30% by weight, said blend furnishing from about 10% to about 25% by weight and polyacrylic acid furnishing from about 75% to about 90% by weight of said solids.

14. A sizing composition for nylon yarn consisting essentially of an aqueous solution of a (1) blend of an aqueous solution of glycollic acid, acrylamide and a condensate of one mole of a polyethylene glycol with a molecular weight of about 400 with about seven moles of ethylene oxide, said glycollic acid comprising from about 70% to about 90% by weight, said acrylamide comprising from about 3% to about 30% by weight and said polyethylene glycol-ethylene oxide condensate comprising from about 3% to about 15% by weight of the total solids in the blend, the total solids in said blend being from about 65% to about 75% by weight thereof, and (2) polyacrylic acid, the total solids in said solution being from about 20% to about 30% by weight, said blend furnishing from about 10% to about 25% by weight and polyacrylic acid furnishing from about 75% to about 90% by weight of said solids.

15. A sizing composition for nylon yarn consisting essentially of an aqueous solution of a (1) blend of an aqueous solution of glycerine, formamide and a condensate of glycollic acid with 4.3 moles of ethylene oxide, said glycerine comprising from about 3% to about 15% by weight, said formamide comprising from about 3% to about 30% by weight and said glycollic acid-ethylene oxide condensate comprising from about 70% to about 90% by weight of the total solids in said blend, the total solids in said blend being from about 65% to about 75% by weight thereof, and (2) polyacrylic acid, the total solids in said solution being from about 20% to about 30% by weight, said blend furnishing from about 10% to about 25% by weight and polyacrylic acid furnishing from about 75% to about 90% by weight of said solids.

16. A sizing composition for nylon yarn consisting essentially of an aqueous solution of a (1) blend of diethylene glycol, a condensate of one mole of diglycollic acid with about two and one-quarter moles of ethylene oxide and a condensate produced by reacting one mole of urea with about one and one-half moles of ethylene oxide, said diethylene glycol comprising from about 3% to about 15% by weight, said glycollic acid-ethylene oxide condensate comprising from about 70% to about 90% by weight and said urea-ethylene oxide condensate comprising from about 3% to about 30% by weight of the total solids in said blend, the total solids in said blend being from about 65% to about 75% by weight thereof, and (2) polyacrylic acid, the total solids in said solution being from about 20% to about 30% by weight, said blend furnishing from about 10% to about 25% by weight and polyacrylic acid furnishing from about 75% to about 90% by weight of said solids.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 2,876,205 March 3, 1959

Charles H. Lighthipe et al.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 7, line 56, for "they arn" read -- the yarn --; column 11, lines 19 and 20, and 27 and 28, for "water-insoluble" read -- water-soluble --.

Signed and sealed this 23rd day of June 1959.

(SEAL)

Attest:

KARL H. AXLINE
Attesting Officer

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Commissioner of Patents