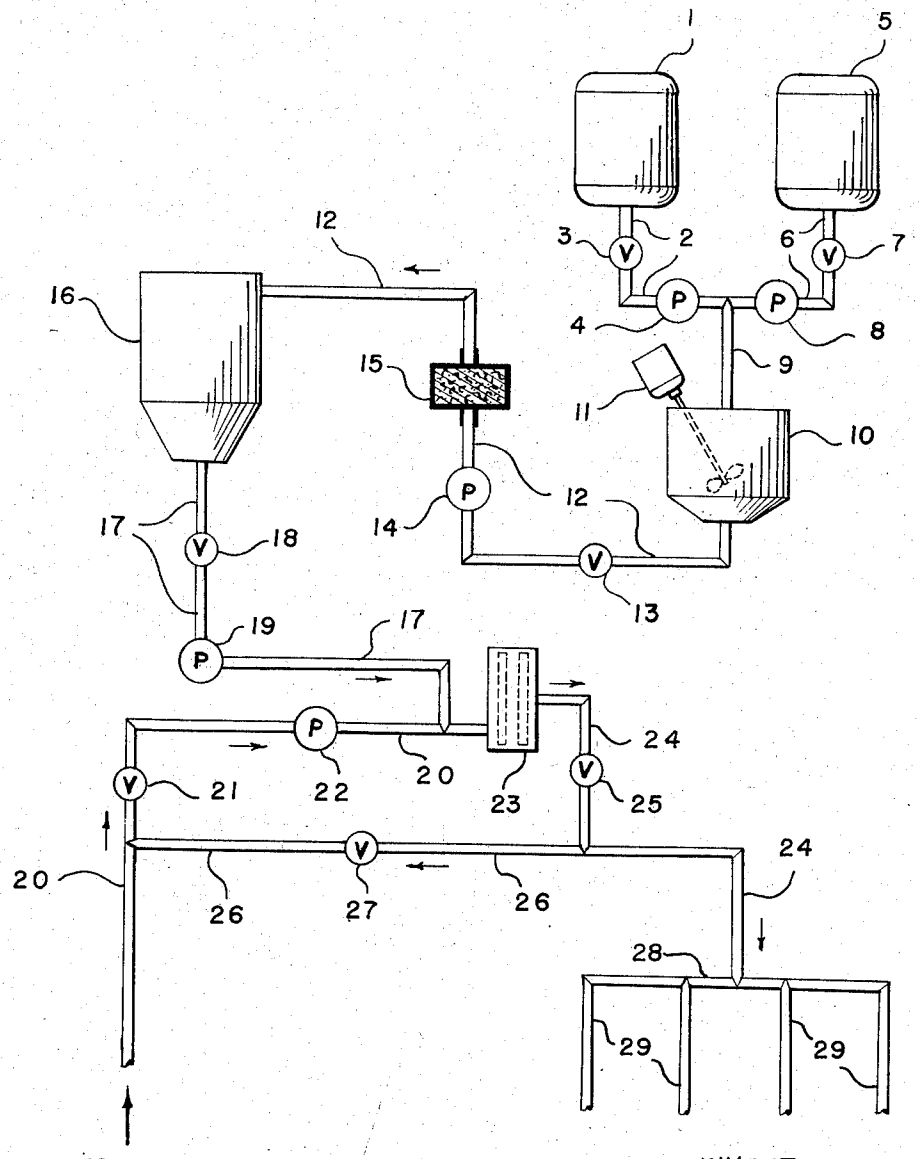
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COMPOSITION FOR AND METHOD OF PIGMENTING VISCOSE

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VISCOSE SUPPLY

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COMPOSITION FOR AND METHOD OF PIGMENTING VISCOSE

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This invention relates to viscose adapted for extrusion into colored yarn and the like. In particular, this invention relates to pigment colored viscose spinning solutions. More particularly, this invention relates to an improved composition for pigmenting viscose and to a method of pigmenting ripened viscose with the improved composition for extrusion into colored yarn and the like.

Colored viscose yarns and the like may be prepared with improved fastness to light and to washing, and with other desirable characteristics by incorporating the coloring matter in the spinning solution prior to extrusion. Viscose so produced, and also yarns from other fiber forming materials, are known as being spun-dyed or solution-dyed yarns. In the production of spun-dyed colored yarn and the like from viscose it has been found advantageous to continuously admix and blend coloring matter into the ripened viscose prior to extrusion and after the ripened viscose, in a spinnable condition, has been delivered from storage or spin tanks to a spinning machine. In this way, the necessity and inconvenience of color blending the ripened viscose spinning solution in batches prior to delivery to the spinning machine for extrusion may be eliminated. Thus, substantial economies may be realized in maintenance and cleaning of equipment, and also in possible losses due to miscolored viscose whenever it may be desired to alter or change the color of yarn being spun. However, in order that consistently similar colored yarn products may be produced from day to day when viscose is being continuously blended with coloring matter, it is important that constant unit proportions of any particular coloring matter be continuously and consistently introduced and blended into unit volumes of the viscose spinning solution during unit periods of extrusion. This is especially important when the viscose being colored is being supplied for continuous extrusion through a plurality of spinning positions, and position-to-position as well as day-to-day variations in the colored yarn product are to be avoided.

Among the various coloring matter which may be employed for coloring viscose spinning solutions, pigments are known to be especially desirable. It is advantageous to employ coloring pigments in the form of aqueous pigment dispersions in order to circumvent the difficulty with which dry pigments of generally fine particle size may be thoroughly blended directly into viscose, in order to attain an intimate, uniform mixture having suitable homogeneity. Generally, aqueous pigment dispersions, sometimes referred to as pigment pastes, are thin, watery fluids of relatively low viscosity and may have various solids contents. However, when attempts are made with conventional metering devices, such as gear pumps, to continuously and directly inject a pigment paste for blending into a ripened viscose spinning solution which is flowing under pressure, difficulties are encountered in admixing constant proportions of the fluid pigment paste into the viscose.

The simplest and most generally employed metering devices for delivering constant volumes of viscous liquids over unit periods of time are gear pumps. Gear pumps, for example, are commonly preferred to extrude viscose and other relatively viscous spinning solutions for spinning since they are adapted to deliver liquids at a substantially constant rate with pulsations in the flow of only relatively minor magnitude. However, due to the slight clearances between the rotors and the casing in a gear pump, they are not well adapted for metering relatively low viscosity or thin fluids against substantial back pressures since considerable slippage of a thinner fluid may occur through the clearance between the rotors and the casing. This is especially so when there is a substantial pressure differential across the pump from its inlet or feed side to its outlet or delivery side. It has been found troublesome, therefore, to employ gear pumps for continuously metering or injecting constant amounts of pigment paste into ripened viscose spinning solutions flowing under pressure, especially when there is an appreciable fluctuating, or even static, variation between the feed pressure of the pigment paste at the pump inlet and the delivery pressure at the pump outlet. Such variations frequently occur over a range of as much as 40 pounds per square inch (p. s. i.), or greater, when pigment paste is injected into a viscose feed line, due to the manner in which viscose is conventionally handled. Obviously, the injection pressure of the pigment paste must meet or exceed the pressure under which the viscose is being handled. When gear pumps are used for this purpose, difficulty in keeping a desired volumetric ratio of pigment paste within tolerant consistent limits in a viscose spinning solution may be experienced due to the heretofore mentioned deficiencies of gear pumps in such situations. Variations of more than about 5% in the pigment content of viscose are known to be visually discernible in colored yarn products produced therefrom. As a consequence, variations greater than about this amount of the pigment content of viscose result in objectionable spinning position-to-position and day-to-day shade differences in the colored yarn product.

Although it is known to pre-blend and disperse pigments in viscose for subsequent admixture and blending with viscose spinning solutions, such a practice is not always satisfactory in that great care must be exerted to avoid the setting-up of the viscose used for dispersing the pigment due to prolonged standing or exposure to elevated temperatures, or both. When this occurs undesirable consequences such as waste and possible equipment or processing stoppages may be experienced. For this reason, no more of the pre-blended pigment dispersed in viscose can be prepared in advance than an amount which may be used within a reasonably short time, even when the pigment dispersion is made up using relatively unripened or green viscose which may take longer to set-up. Hence, larger quantities of pigment dispersed in viscose, which might be sufficient for extended periods of spinning, may not conveniently be prepared in advance. In addition, any excess or unused amount of pigment dispersed in viscose which may occur may not readily be stored for later use due to the tendency of viscose to set-up upon standing.

The present invention provides for an improved pigment mixture which may readily be precisely and continuously introduced with a constant delivery device, such as a gear pump, into ripened viscose under pressure, prior to extrusion, to produce colored yarns and the like. The improved pigment mixture of the present invention may be prepared and stored for considerable periods extending over several months in advance of requirements without harmful consequences. Excess prepared amounts

may likewise be saved until their later use is occasioned. In addition, the present invention advantageously provides for an improved method of continuously pigmenting ripened viscose spinning solutions flowing under pressure to a plurality of spinning positions. The method minimizes spinning position-to-position shade differences in the colored yarn product and colored yarn having substantially consistent color characteristics may be spun over substantial periods of time from a plurality of spinning positions.

The composition for pigmenting viscose in accordance with the present invention comprises a yarn coloring pigment homogeneously dispersed in a stable, viscous liquid, miscible and compatible with viscose, which advantageously may be a glucose containing syrup, for example, a syrup resulting from the hydrolysis of starch, such as corn syrup. The pigment, in a finely divided state, is initially prepared in an aqueous dispersion or pigment paste. The pigment paste is then homogeneously mixed with sufficient amounts of a glucose containing syrup to attain a stable, homogeneous, non-setting mixture having a constant consistency and which is adapted to be metered from a constant delivery device, such as a gear pump, with not more than about 5% volumetric variation in delivery over a unit period of time when pumping against delivery pressures at the pump outlet varying over a range of at least about 40 p. s. i. from the pressures at the pump feed or inlet. Viscose flowing under pressure may be continuously and consistently pigmented in accordance with the present invention by injecting from a constant delivery gear pump sufficient coloring amounts of the pigment paste/syrup composition of the present invention into the viscose and subsequently physically blending the viscose with the paste/syrup composition. Viscose spinning solutions thus pigmented may subsequently be extruded through a plurality of spinning positions over extended periods of time into colored yarn products having minimized day-to-day and spinning position-to-position shade differences.

Other additional objects and advantages of the present invention will be apparent in the following description and the drawing which schematically represents the method of the present invention for blending a viscose spinning solution with the improved pigmenting composition for extrusion into colored yarn and the like.

With reference to the drawing, a finely ground coloring pigment, prepared in aqueous dispersion, may be stored in a tank **1** as pigment paste. The glucose containing syrup, which advantageously may be a corn syrup of at least about 38° Bé., may be stored in a similar tank **5**. To prepare the pigment paste/syrup composition, a measured amount of pigment paste is delivered by a pump **4** through a conduit **2**, valve **3** and conduit **9** into a mixing tank **10**. A quantity of syrup is likewise withdrawn from the tank **5** through a conduit **6** and valve **7** by a pump **8** and is transmitted through the conduit **9** to the mixing tank. The quantity of syrup is determined by the amount and character of pigment paste being used and the nature of the syrup being employed in order to obtain a paste/syrup composition having the desired characteristics and consistency. For example, when a corn syrup having a concentration of at least about 38° Bé., is employed, especially when the concentration is about 42° Bé., a satisfactory paste/syrup composition can usually be prepared by mixing at least about 2½ parts by volume of the syrup with each part by volume of the pigment paste. In the mixing tank **10** the pigment paste and syrup are homogeneously mixed by a mixer **11** which may be of any conventional type suited for the purpose. The resulting stable, homogeneous paste-syrup composition, containing uniformly dispersed pigment, is passed through a conduit **12** by a pump **14** into a storage tank **16** where it is stored at a room temperature between about 60° and about 80° F. ready for use. A valve **13** may be positioned in the conduit **12** and a filter **15** may be employed to filter the paste/syrup mixture passing through the conduit **12** before it is collected in the storage tank **16**. If desired, filtration may be else where performed, as for example, upon withdrawal from the storage tank, or it may not be necessary.

As mentioned, the paste/syrup composition is so proportioned that it may be delivered at a room temperature from a constant delivery gear pump, adapted for metering viscous liquids, with not more than about 5% variation in volumetric delivery with pressures at the pump outlet varying over a range of at least about 40 p. s. i. from those at the pump inlet. Such a range, for example, would be experienced when delivering a paste/syrup composition under delivery pressures, or back pressures, at the pump outlet varying between about 1 and about 41 p. s. i. after the composition has been fed to the pump inlet under a pressure of about 1 p. s. i. The paste/syrup composition maintained at a room temperature and without requiring agitation to keep the pigment dispersed, may be fed by gravity from the storage tank **16** through a conduit **17** and valve **18** to a constant delivery gear pump **19**. The gear pump **19**, operating at a desired rate, consistently meters the desired constant volume of the paste/syrup composition through the conduit **17** for injection into a flow of ripened viscose spinning solution being transmitted under pressure through a feed conduit **20** which, for example, may supply a spinning machine. The viscose spinning solution, obtained from a supply source (not shown), is transmitted under pressure through the feed conduit **20** and a valve **21** by a metering gear pump **22** in desired proportionate volumes. If desired, the paste/syrup composition may be injected into the viscose ahead of the metering pump **22** in the conduit **20**. The viscose containing the paste/syrup composition is blended in a blender **23** which may be of any suitable type, for example, the disc blender **23** which intimately combines the material between closely spaced rotating discs. After blending, the pigmented viscose passes through a conduit **24** and a valve **25** to a spinning solution supply manifold **28** from which it is delivered through a plurality of conduits **29** to be extruded at a plurality of spinning positions (not shown). A portion of the pigmented viscose, after blending, may be diverted from the conduit **24** through a conduit **26** and a valve **27** to be fed back and recirculated with fresh viscose from the supply source. The feed-back ratio of the blended viscose may advantageously be at about 50% although any desired portion of the blended viscose, or none at all, may be fed back for recirculation. Viscose spinning solution may be continuously pigmented according to this method to produce consistently colored yarn and the like.

The glucose containing syrups in which the pigment may be dispersed for blending with ripened viscose spinning solution in accordance with the present invention are those which are sufficiently viscous to produce a pigment composition which may successfully be handled at about room temperature by a constant delivery gear pump as heretofore explained. Although syrups of varying consistency may be employed in suitable paste/syrup compositions, it is generally advantageous to use syrups having a concentration of at least about 38° Be., according to the method for determining the Baumé equivalent of a liquid which is commonly employed in the syrup industry. When syrups of such a concentration are employed the ratio of syrup to pigment paste required is not generally high, and consequently the volumes of a paste/syrup composition which must be injected into viscose for solution dyeing are not excessively large because of there being a relatively low pigment content in the paste/syrup composition. Generally, a ratio in the approximate neighborhood of about three parts by volume of syrup to about each part of volume of pigment paste is found to be desirable. The syrups are generally water-including complex carbohydrate materials which may contain other mono-saccharides, di-saccharides, higher saccharides or other carbohy-

drates in addition to glucose. Advantageously, a syrup product resulting from the partial or incomplete hydrolysis of starch is employed. Preferably, hydrolyzed corn starch, known as corn syrup, is employed although syrups from other starches including potato starch and other grain and vegetable starches also may be suitable. These syrups are frequently generally known as glucose syrups. Corn syrup, which contains some water in addition to glucose, maltose, dextrins and other constituents, is available as a crystal clear, uncrystallizable liquid over a range of relatively high concentrations and viscosities which are particularly useful in accordance with the present invention. In many cases, however, less highly refined corn syrups which are clear but not colorless may be satisfactorily utilized. A corn syrup having a concentration of at least about 38° Bé., and a dextrose equivalent (D. E.) of between about 35 and about 60 may, for example, be advantageously employed. As it is commonly understood, the dextrose equivalent of a syrup product resulting from a partial or incomplete hydrolysis of a starch, for example corn syrup from corn starch, is a measure of the degree of conversion of the starch to sugar or, explained otherwise, the extent of hydrolysis which the starch has undergone. The dextrose equivalent is ordinarily defined as the percentage of reducing sugar substances in the syrup calculated as dextrose on the dry weight basis. It may be expressed numerically as percentage D. E. or D. E. Ordinarily starch syrups of lower conversion, having a D. E. beneath about 35, while probably not entirely useless, are not considered to be as desirable for the purposes of this invention as are the more highly converted syrups. Lower conversion products which have a greater content of incompletely hydrolyzed and higher molecular weight starch constituents generally appear to be not as suitable as the higher conversion syrups. Starch syrups having a D. E. greater than about 60 may be considered as sugars and often have a tendency to crystallize. This behavior renders them not always suitable for preparing paste/syrup compositions due to the possibility of plugging spinnerettes with crystallized particles. A corn syrup having a D. E. between about 40 and about 45 and a concentration of at least about 41° Bé. and advantageously at about 42° Bé. is especially desirable. Particularly useful is a corn syrup having a D. E. of about 43 and a concentration of about 42° Bé. and which may contain various sugars in an approximate amount of about 55% by weight, of which approximately 34% by weight is glucose, and water in an amount not in excess of approximately about 20% by weight. Other glucose containing syrups may be also employed besides those which are starch hydrolysis products. For example, syrups having a base of invert sugar, which may result from the hydrolysis of a di-saccharide such as sucrose, may be employed. Accordingly, honey, molasses, sugar-beet syrup, sugar-cane syrup and like syrups may be useful with the present invention as are other glucose containing syrups having sufficiently high relative viscosity to thicken pigment paste sufficiently in the heretofore explained manner. Frequently, as is the case with corn syrup, an increasing specific gravity of the syrup above that of water and a higher concentration as evidenced by its Baumé equivalent are characteristics which often reliably signify an increasing glucose content along with an associated higher viscosity. As mentioned, syrups having a concentration of at least about 38° Bé. are usually more desirable. Generally, thicker syrups produce satisfactory compositions with lower syrup to paste ratios. However, thinner syrups may also be employed if it is suitable to operate with a relatively higher syrup to paste ratio. Syrups concentrated much above 43° Bé. may be difficult to handle at room temperatures due to their extremely viscous nature. However, they may be satisfactorily employed for paste/syrup compositions if suitable means are available for handling them. Oftentimes, for example, thicker syrups may be heated during handling while being mixed with the pigment paste to produce a composition manageable at a room temperature in the usual manner.

The employment of glucose containing syrups to thicken the pigment pastes in accordance with the present invention is highly advantageous in many particulars. Many syrups, in addition to corn syrup, are obtainable which are clear or colorless, if desired. They are also available free of included particles of a nature which could not be tolerated in the manufacture of viscose yarn. They present no special filtration difficulties and, in addition, the commonly available glucose containing syrups are inert to viscose and are completely miscible and compatible therewith. Hence, they may be incorporated in a viscose spinning solution without fear of harmful results. They are generally stable and inert upon standing to any changes in viscosity or composition because of intrinsic physical or chemical activity. Furthermore, the pigment compositions of the present invention composed with glucose containing syrups are stable, homogeneous, and have little tendency to settle on standing. Generally, they do not require agitation during use to maintain the pigment dispersion. Also, these compositions may more advantageously be handled by constant delivery gear pumps because they retain their consistency and exhibit a true viscosity without tending to assume thixotropic pecularities. Many thickening agents may be deficient in these respects. For example, carboxymethylcellulose type materials, which are often used for thickening purposes, are not completely satisfactory for thickening pigment paste. They not only filter with difficulty and may contain particles of undissolved fiber and the like which tend to clog spinnerettes, but pigments dispersed in carboxymethylcellulosic compositions seem to possess a static viscosity and thin out to an undesirable degree upon being handled under pressure.

The pigments which may be blended into viscose in accordance with the present invention are those commonly employed for spun-dyed or solution dyed viscose and other spinning solutions. They may be inorganic or organic in nature or may be organic salts and the like and also may be mixtures of various pigment, or other coloring, compounds. These pigments are used in a finely divided state in order to facilitate their being passed through the small orifices of spinnerettes and similar yarn extruding devices. Ordinarily, for example, about 99% of the particles in these pigments may have a size not in excess of about one micron with the balance of the particles being not in excess of about five microns in size. As mentioned, the pigment may be dispersed in water, or other fluids, to form pigment paste possibly with the aid of a dispersing agent. A pigment paste may contain, for example, about 20% by weight of dispersed solids although varying solids contents may be effected.

Viscose spinning solutions which may be pigmented in accordance with the present invention may be of any spinnable type desired. For example, regular bright, dull delustered or partially delustered viscose having varying contents of cellulose and other ingredients may be pigmented for extrusion into colored yarn and the like. As mentioned, the viscose to be pigmented is advantageously at a desired level of ripeness and ready for spinning in order to be pigmented in accordance with the present invention.

In order to further illustrate the invention but without being limited thereto the following example is given:

Example

About one part by volume of a pigment paste containing about 20% by weight of dry copper phthalocyanine (Monastral Blue) pigment in aqueous dispersion was homogeneously mixed with about three parts by volume of about 42° Bé. corn syrup having a D. E. of about 43 and containing about 55% by weight of various sugars, of which about 34% by weight were glucose, and about 20% by weight of water to form a stable paste/syrup

composition that would not separate on standing. The composition was filtered through cotton batting and muslin and was tested for delivery characteristics using a gear type metering pump (such as a commercially available Zenith pump). When feeding the paste/syrup composition to the pump by gravity, it was found possible to deliver a substantially constant volume per unit time of the composition from the gear pump against delivery pressures at the pump outlet ranging between about 20 and about 60 p. s. i. The variation in volumetric delivery per unit time under such conditions was not more than about 1% by volume. The paste/syrup composition, needing no agitation to retain the pigment in homogeneous dispersion, was fed by gravity to a gear type metering pump for injection into a flow of ripened viscose spinning solution being transmitted in a feed line under about 80 p. s. i. average pressure. About one part by volume of the paste/syrup composition was consistently and continuously metered into about every 250 parts by volume of fresh viscose from a viscose supply source. After injection, the paste/syrup composition and the viscose were intimately blended using a disc type blender. About 50% by volume of the pigment blended viscose was fed back to the fresh viscose spinning solution being supplied and recirculated therewith. The unrecirculated pigment blended viscose was supplied to a plurality of spinning positions for extrusion. A plurality of colored yarns were thus spun over an extended period of time. The colored yarn thereby produced was commercially acceptable. Spinning position-to-position shade differences were negligible and there were no visually noticeable day-to-day differences between the shade of yarn from any position spun at the beginning of the period from that of yarn spun at its conclusion.

The amount of pigment employed for blending with the viscose spinning solution depends, of course, upon the particular pigment being employed and the depth to which it is desired to color the resulting yarn product. Likewise, the proportions necessary to compound the pigmenting compositions in accordance with the present invention vary with the type of pigment and pigment paste being employed, the particular glucose containing syrup being utilized and the ultimate characteristics which are desired in the colored yarn product. Generally, by experimentation, a particular pigment paste/syrup composition is empirically compounded in order to be adapted for satisfactory delivery from a metering device as heretofore explained. Due to the varying nature of the ingredients and the multiple influences which may come into play, it is often difficult to accurately predict suitable generalized formulations for paste/syrup pigmenting compositions in accordance with the present invention.

Since certain changes in practicing the invention may be made without departing from its spirit or scope, it is to be understood that all the foregoing description be interpreted as merely illustrative and in no sense limiting of the invention, as particularly pointed out and defined in the appended claims.

What is claimed is:

1. Pigmenting composition for viscose spinning solution for subsequent extrusion into colored yarn and the like consisting essentially of not more than about one part by volume of an aqueous yarn coloring pigment dispersion containing not more than about 20% by weight of dispersed solids homogeneously mixed with at least about 2½ parts by volume of an uncrystallizable liquid corn syrup having a concentration of at least about 38° Baumé and a dextrose equivalent between about 35 and about 60.

2. Pigmenting composition for viscose spinning solutions for subsequent extrusion into colored yarn and the like consisting essentially of about one part by volume of an aqueous yarn coloring pigment dispersion containing no more than about 20% by weight of dispersed solids mixed with about 3 parts by volume of an uncrystallizable liquid corn syrup having a concentration of about 42° Baumé and a dextrose equivalent between about 40 and about 45.

3. Method for pigmenting a ripened viscose spinning solution flowing under pressure for subsequent extrusion into colored yarn and the like comprising homogeneously mixing not more than about one part by volume of an aqueous yarn coloring pigment dispersion containing no more than about 20% by weight of dispersed solids with at least about 2½ parts by volume of a corn syrup having a concentration of at least about 38° Baumé and a dextrose equivalent between about 35 and about 60 to produce a mixture adapted to be delivered from a constant delivery gear pump with not more than about 5% volumetric variation per unit time when being pumped against delivery pressures at the pump outlet varying over a range of at least about 40 p. s. i. from pressures at the pump inlet; injecting the mixed pigment syrup with a constant delivery gear pump into said ripened viscose spinning solution flowing under pressure; then blending the mixed pigment syrup and viscose prior to extrusion.

4. Method for pigmenting a ripened viscose spinning solution flowing under pressure for subsequent extrusion into colored yarn and the like comprising homogeneously mixing about one part by volume of an aqueous yarn coloring pigment dispersion containing no more than about 20% by weight of dispersed solids with about 3 parts by volume of a corn syrup having a concentration of about 42° Baumé and a dextrose equivalent between about 40 and about 45; injecting the mixed pigment syrup with a constant delivery gear pump into said ripened viscose spinning solution flowing under pressure; then blending the mixed pigment syrup and viscose prior to extrusion.

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