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MANUFACTURE OF YARNS AND THE LIKE

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No Drawing. Application June 27, 1944, Serial No. 542,431, which is a division of application Serial No. 389,325, April 19, 1941, now Patent No. 2,359,749, dated October 10, 1944. Divided and this application May 28, 1947, Serial No. 751,136

9 Claims. (Cl. 18—54)

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This invention relates to improvements in the manufacture of synthetic yarns, filaments, films and the like. More particularly, the invention relates to improvements in the spinning operation employed in the manufacture of synthetic yarns, filaments, films and the like from viscose. This application is a division of my copending application Serial No. 542,431, filed June 27, 1944, now abandoned which is in turn a division of my application Serial No. 389,325, filed April 19, 1941, now Patent 2,359,749, dated October 10, 1944.

In the manufacture of synthetic shaped articles such as yarns, filaments, films and the like from viscose, a solution is generally first prepared of the viscose to comprise the article. The article is formed or spun by extruding the solution through a spinneret or jet, usually made of metal, having a minute opening or openings into a coagulating or precipitating medium. During the spinning operation obstructions tend to form in the minute openings in the spinneret or jet and they become fouled or clogged. This results, in the case of yarn or filament spinning, in a lack of uniformity in the filament sizes and weak and broken filaments in the finished product and, in the case of producing films, in a lack of uniformity in thickness. In order to avoid these difficulties it is the present practice to inspect the spinnerets or jets at short intervals of time and replace those in which the openings have become fouled. This interrupts the continuity of the spinning operation and causes delays which are time-consuming and costly.

This invention has for its principal object to provide improvements in the spinning operation employed in the manufacture of synthetic shaped articles such as yarns, filaments, films and the like.

Other objects of the invention will be apparent from the following description and appended claims.

It has now been found that fouling of the spinnerets and the need for frequent inspection and replacement and the lack of uniformity in yarns, filaments and films can be largely eliminated by extruding the spinning solution in the presence of a small amount of a surface active, non-ionogenic organic compound of essentially aliphatic character and comprising at least one hydrophobic group having a hydrocarbon chain of at least 8 carbon atoms and a hydrophilic polyoxyethylene content of at least 8 oxyethylene units, this content in any particular case being sufficient to render the compound water-soluble so that it is readily soluble in the spinning bath

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or in the spinning solution, all of the chain-forming atoms in the compound consisting of carbon and oxygen. Generally, the greater the extent to which the compound is hydrophobic, the larger the number of oxyethylene units that are required to render the compound sufficiently soluble for the purposes of the invention.

In general, the compounds contemplated by the invention are the polyethylene glycol ethers of organic compounds having at least one functional aliphatic hydroxyl group and containing at least one hydrophobic group comprising a hydrocarbon chain of at least 8 carbon atoms, such as alcohols and polyhydric alcohols, hydroxy carboxylic acids, hydroxy esters, epoxy alcohols and hydroxy epoxy esters; polyethylene glycol esters of compounds having at least one functional aliphatic carboxyl group and containing at least one hydrophobic group comprising a hydrocarbon chain of at least 8 carbon atoms, such as fatty acids, polycarboxylic acids, hydroxy fatty acids, and epoxy fatty acids; polyethylene glycol acetals of organic compounds having at least one functional aliphatic aldehyde group and containing at least one hydrophobic group comprising a hydrocarbon chain of at least 8 carbon atoms, such as aliphatic aldehydes, and polyethylene glycol ether-esters, ether-acetals, ester-acetals or ether-ester-acetals of organic compounds having various combinations of hydroxyl, aldehyde, and carboxyl groups and containing at least one hydrophobic group comprising a hydrocarbon chain of at least 8 carbon atoms, such as hydroxy fatty acids, polyhydroxy fatty acids, aldoses, and -uronic acids. The compounds are non-ionogenic, relatively chemically unreactive, substantially stable under spinning conditions, and contain no functional groups other than ether (oxo), ester, acetal, aliphatic hydroxyl and carbonyl groups which are substantially stable under the conditions of spinning. The stability herein referred to is such stability that the compounds, prior to their complete decomposition in the spinning solution or the spinning bath, will exert their favorable influence in preventing clogging of the openings in the spinnerets and in thereby maintaining uniformity of filament size.

The following are examples of specific compounds that may be used in carrying out this invention:

Caprylic acid esters of polyoxyethylene glycols
Pelargonic acid esters of polyoxyethylene glycols
Capric acid esters of polyoxyethylene glycols
Lauric acid esters of polyoxyethylene glycols

Myristic acid esters of polyoxyethylene glycols
Palmitic acid esters of polyoxyethylene glycols
Margaric acid esters of polyoxyethylene glycols
Stearic acid esters of polyoxyethylene glycols
Oleic acid esters of polyoxyethylene glycols
Ricinoleic acid esters of polyoxyethylene glycols
Hydroxy stearic acid esters of polyoxyethylene glycols
Octyl ethers of polyoxyethylene glycols
Decyl ethers of polyoxyethylene glycols
Dodecyl ethers of polyoxyethylene glycols
Tetradecyl ethers of polyoxyethylene glycols
Hexadecyl ethers of polyoxyethylene glycols
Octadecyl ethers of polyoxyethylene glycols
Cetyl ethers of polyoxyethylene glycols
Montanyl ethers of polyoxyethylene glycols
Oleyl ethers of polyoxyethylene glycols
Polyoxyethylene glycol ethers of ethylene glycol monoacyl[1] esters
Polyoxyethylene glycol ethers of diethylene glycol monoacyl[1] esters
Polyoxyethylene glycol ethers of polyethylene glycol monoacyl[1] esters
Polyoxyethylene glycol ethers of propylene glycol monoacyl[1] esters
Polyoxyethylene glycol ethers of glycerol monoacyl[1] esters
Polyoxyethylene glycol ethers of glycerol diacyl[1] esters
Polyoxyethylene glycol ethers of polyglycerol acyl[1] partial esters
Polyoxyethylene glycol ethers of erythritol acyl[1] partial esters
Polyoxyethylene glycol ethers of pentaerythritol acyl[1] partial esters
Polyoxyethylene glycol ethers of hexitol acyl[1] partial esters
Polyoxyethylene glycol ethers of mannitol acyl[1] partial esters
Polyoxyethylene glycol ethers of sorbitol acyl[1] partial esters
Polyoxyethylene glycol ethers of hexitan acyl[1] partial esters
Polyoxyethylene glycol ethers of mannitan acyl[1] partial esters
Polyoxyethylene glycol ethers of sorbitan acyl[1] partial esters
Polyoxyethylene glycol ethers of hexide acyl[1] partial esters
Polyoxyethylene glycol ethers of mannide acyl[1] partial esters
Polyoxyethylene glycol ethers of sorbide acyl[1] partial esters
Polyoxyethylene glycol ethers of cyclotol acyl[1] partial esters
Polyoxyethylene glycol ethers of inositol acyl[1] partial esters
Polyoxyethylene glycol ethers of monosaccharide acyl[1] partial esters
Polyoxyethylene glycol ethers of oligosaccharide acyl[1] partial esters

More specific combinations are:

Polyoxyethylene glycol ethers of mannitan monolaurate
Polyoxyethylene glycol ethers of sorbitan monolaurate
Polyoxyethylene glycol ethers of sorbitan monoleate
Polyoxyethylene glycol ethers of mannitan monomyristate

[1] The acyl esters may be the esters of fatty acids having 8 carbon atoms in a straight chain, such as caprylic, pelargonic, capric, lauric, myristic, palmitic, margaric, stearic, oleic, ricinoleic, and hydroxy stearic.

Polyoxyethylene glycol ethers of mannitan monopalmitate
Polyoxyethylene glycol ethers of mannitan monostearate
Polyoxyethylene glycol ethers of mannitan monooleate
Polyoxyethylene glycol ethers of triricinolein
Polyoxyethylene glycol acetals of lauric aldehyde

The polyethylene glycol group or groups may be connected in the same compound by either or both ether and ester groups depending upon the parent substances from which they are derived and the method of producing the compounds therefrom. For example, if ricinoleic acid, hydroxy stearic acid or other bi-functional compounds are reacted with ethylene oxide, part of the oxyethylene units may replace the hydrogen of the hydroxyl group while another part may replace the hydrogen of the carboxyl group, thereby producing polyoxyethylene ether-esters.

It should be noted that all of the oxyethylene units may be in a single chain or they may be present in the compound in the form of two or more chains, the latter situation being common when the compound is produced by reacting ethylene oxide with a parent substance having at least two functional groups whether hydroxyl, aldehyde, carboxyl or any combination of these three. It is only essential that the total number of oxyethylene units in the one or more chains add up to a total of at least eight and sufficient in any particular case to render the compound soluble in the viscose or in the acid coagulating bath. Some or all of the oxyethylene units or chains of such units may have either or both of their ends connected to the remainder of the compound through ether, ester or acetal linkages.

The common methods of making the compounds with which the invention is concerned generally result in the formation of mixtures of combounds having a greater or less number of oxyethylene units per molecule and analytical determinations of such complex mixtures indicate the average number of such units per molecule throughout the mass of the compound analyzed. For this reason the compounds employed in accordance with this invention may comprise molecules containing less than 8 oxyethylene units in admixture with others containing 8 or more such units. Accordingly, in the specification and the claims the definition of these compounds is intended to include not only those having a uniform and constant constitution throughout but also such complex mixtures as have an effective portion containing at least 8 oxyethylene units per molecule.

The various surface active compounds may be produced in any suitable manner. Generally, they may be produced by reacting ethylene oxide with the selected fatty acid, alcohol, hydroxy fatty acid, hydroxy fatty acid ester or related compound within the scope of the general class described above. For example, the reaction products of ethylene oxide with sorbitan monolaurate may be prepared by introducing sorbitan monolaurate with a small amount of sodium methylate as a catalyst into a stirring autoclave and raising the temperature to 100° C., gradually adding ethylene oxide to this mixture and maintaining the temperature between 105° C. and 110° C. For reasons stated above, the degree to which the reaction with the ethylene oxide is carried out depends upon the compound with which it is reacted and the character of the

spinning solution or precipitating bath in which it is to be used. The reaction is carried out to such a degree that the reaction products are soluble in the spinning bath or spinning solution in which they are used.

The invention is applicable in forming regenerated cellulose shaped articles by extruding viscose solution into an acid precipitating bath, especially those containing sulfuric acid, and is found particularly advantageous where the precipitating bath contains a high concentration of metallic salts, especially zinc, in concentrations of the order of 3% to 10%.

In carrying out the invention a small amount of the surface active material of the order of 0.01 to 0.05% may be dissolved in the spinning solution and the spinning solution is extruded into a precipitating medium. Where a liquid precipitating medium or bath is used, a small amount of the surface active material of the same order may be dissolved in the precipitating medium or may be added to both the spinning solution and the precipitating medium.

The following examples are illustrative of the invention:

Example 1.—A multifilament regenerated cellulose yarn is formed by extruding viscose solution through a spinneret into an aqueous sulfuric acid spinning bath containing a high concentration of zinc sulfate of the order of 5% and between 0.02 and 0.03% of organic compounds comprising a mannitan monolaurate having at least 1 hydroxy polyoxyethylene group per molecule and an average oxyethylene content of 20 oxyethylene units per molecule.

Example 2.—A multifilament regenerated cellulose yarn is formed by extruding viscose solution through a spinneret into an aqueous sulfuric acid spinning bath containing a high concentration of zinc sulfate of the order of 5% and between 0.02 and 0.03% of organic compounds comprising a mannitan monopalmitate having at least 1 hydroxy polyoxyethylene group per molecule and an average oxyethylene content of 20 oxyethylene units per molecule.

Example 3.—A multifilament regenerated cellulose yarn is formed by extruding a viscose solution through a spinneret into an aqueous sulfuric acid spinning bath containing a high concentration of zinc sulfate of the order of 5% and between 0.02 and 0.03% of organic compounds comprising an acetal of lauric aldehyde having an average oxyethylene content of 20 oxyethylene units per molecule.

Example 4.—A multifilament regenerated cellulose yarn is formed by extruding a viscose solution containing about 0.03% of an acetal of lauric aldehyde having an average of 10 oxyethylene units per molecule through a spinneret into an aqueous sulfuric acid spinning bath containing a high concentration of zinc sulfate of the order of 5%.

It was found that in carrying out the spinning operation in accordance with the present invention it is necessary to change less than 50% as many spinnerets as where ordinary methods of spinning are followed. Where multifilament yarn is formed the filaments are of uniform cross-section and the yarn is of generally improved quality.

While preferred embodiments of the invention have been shown, it is to be understood that changes and variations may be made without departing from the spirit and scope of the invention as defined by the appended claims.

I claim:

1. The method of forming artificial filaments comprising extruding viscose into an aqueous acid coagulating liquid in the presence of a small amount of a dissolved, substantially stable, surface-active non-ionogenic acetal of a polyethylene glycol containing a hydrophobic group comprising a hydrocarbon chain of at least 8 carbon atoms and containing at least 8 oxyethylene units per molecule and a sufficient number of such units in excess of eight to render the compound substantially soluble in the viscose solution.

2. The method of claim 1 in which the acetal is present in the viscose spinning solution.

3. The method of claim 1 in which the acetal is present in the coagulating liquid.

4. The method of claim 1 in which the acetal is present in both the viscose and coagulating liquid.

5. The method of claim 1 in which the acetal is that derived from lauric aldehyde.

6. A viscose spinning solution containing a small amount of a dissolved, substantially stable, surface-active non-ionogenic acetal of a polyethylene glycol containing a hydrophobic group comprising a hydrocarbon chain of at least 8 carbon atoms and containing at least 8 oxyethylene units per molecule and a sufficient number of such units in excess of eight to render the compound substantially soluble in the viscose solution.

7. An aqueous liquid for coagulating viscose solutions, said liquid comprising an acid and a small amount of a dissolved, substantially stable, surface-active non-ionogenic acetal of a polyethylene glycol containing a hydrophobic group comprising a hydrocarbon chain of at least 8 carbon atoms and containing at least 8 oxyethylene units per molecule and a sufficient number of such units in excess of eight to render the compound substantially soluble in the viscose solution.

8. The viscose of claim 6 in which the acetal is that derived from lauric aldehyde.

9. The coagulating liquid of claim 7 in which the acetal is that derived from lauric aldehyde.

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