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MANUFACTURE OF VISCOSE RAYON

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11 Claims. (Cl. 18—54)

1

The present invention relates to the manufactuhe of viscose rayon and more particularly to a process involving the addition of surface active agents to the viscose solution and to the acid coagulating bath in order to improve the characteristics of the finished product and to improve the spinning conditions generally.

It is known to add anion active compounds such as highly sulphonated non-mineral oils of the type sold under the trade-name Prestabit Oil to viscose solutions in order to inhibit turbidity or milkiness in the rayon. (See U. S. Patents Nos. 1,925,192 and 1,936,479.) While these compounds are effective in this respect, they, as well as all anion active materials have a tendency to promote clogging of spinneret orifices. It was not until the use of cation active compounds (see U. S. Patent No. 2,125,031) was commenced that spinneret clogging was substantially prevented.

Thus, in order to improve both the yarn and the spinning conditions it has been necessary to add an anion active compound to the viscose solution and a cation active compound to the acid spinbath. This is obviously objectionable because the two classes of materials come in contact with one another during the spinning operation and have a tendency toward compound formation and mutual precipitation. In addition, the anion active compound in the viscose may counteract or retard the effectiveness of the cation active compound in the spinbath with respect to spinneret incrustation.

Accordingly, on a theoretical basis it would be more feasible to use either one or more anion active compounds or one or more cation active compounds in both the viscose solution and spinbath. However, since it is known that anion active materials cannot be used in the spinbath for practical reasons, it is necessary that the material common to both the viscose solution and the spinbath be cationic in character.

It is therefore the object of the present invention to provide a cation active compound that serves both the purpose of inhibiting milkiness in the viscose yarn and reducing spinneret incrustation in the spinbath.

Another object of this invention is to use small amounts of a cation active compound either in the viscose solution whereby it will get into the spinbath during the spinning operation, or add it directly to the spinbath in addition to adding it to the viscose.

A further object of the invention is to utilize a particular class of cation active compounds that are good dispersants and are sufficiently soluble

2

and stable in both alkali and viscose to prevent milkiness in the yarn while at the same time are sufficiently soluble and stable in the acid spinbath to prevent spinneret incrustation.

It has now been determined that if a long chain aliphatic primary amine is caused to react with an alkylene oxide such as ethylene oxide to form a tertiary amine, its properties can be modified to the extent of rendering it suitable for preventing or inhibiting milkiness in the yarn while at the same time reducing spinneret incrustation. By controlling the length of the alkylene oxide chains, derivatives can be prepared that are soluble and stable in both the alkaline viscose solution and the acid spinbath. The necessary length of the alkylene oxide chains is dependent on the length of the alkyl group in the amine because the hydrocarbon group becomes more hydrophobic as the chain length increases whereas the alkylene oxide chain becomes more hydrophilic as the chain length increases. Therefore, it is only necessary to control the length of the alkylene oxide chain so that it predominates over the alkyl group in the amine whereby the condensate is rendered sufficiently soluble in an aqueous solution to be effective for the purposes here involved.

Compounds of this type are known in the trade as Ethomeens and reaction products of long chain aliphatic primary amines with ethylene oxide, the reaction being represented generally as follows:

$$RNH_2 + (x+y)C_nH_{2n}O \longrightarrow RN\begin{matrix}(C_nH_{2n}O)_xH\\(C_nH_{2n}O)_yH\end{matrix}$$

In the above formula, R represents an aliphatic chain having from 8 to 24 carbon atoms therein and preferably from 12 to 14 carbon atoms. x or y is at least one, and $x+y$ is the total number of alkylene oxide chains which may be as low as 5 without any definite restriction on the upper limit although 12 to 18 or about 15 have been found to be quite effective, and n is a small whole number from 2 to 4, both inclusive.

Specific Ethomeens which have proved very effective in preventing milkiness and spinneret incrustation are known as Ethomeen C/25 and Ethomeen S/25. Resolving these into their structural formula to the extent possible they are:

$$RNH_2 + 15(C_2H_4O) \longrightarrow RN\begin{matrix}(C_2H_4O)_xH\\(C_2H_4O)_yH\end{matrix}$$

The length of the aliphatic chain R depends on the oil from which the parent amine is prepared,

which in the case of C/25 is coco and S/25 is soya. In both cases 15 moles of ethylene oxide are added to the amine. The ethylene oxide apparently separates fortuitously into two chains designated as x and y to form the tertiary amine. It has been demonstrated that the second mole of ethylene oxide is more strongly attracted to the amino nitrogen than it is to the hydroxyl group so that a bishydroxy ethyl alkyl amine is formed. After a total of 5 moles of ethylene oxide had been added to the primary amine it was determined that it had been over 99% converted into a tertiary amine. Therefore, theoretically, the reaction product may contain as low as 5 moles of ethylene oxide provided the solubility is sufficient in alkali and acid although thus far it has been determined for optimum results, about 15 moles of ethylene oxide are required for the reaction.

In accordance with the invention the long chain aliphatic tertiary amine-alkylene oxide condensate is added in small quantities, e. g., 0.12% to 0.24% based on the weight of cellulose to the viscose and, if required, lesser amounts to the acid spinbath based on the weight of the spinbath, of course taking the necessary precautions to insure an even and uniform admixture therewith. In practice, it has been determined that certain advantages are realized if the condensate is added in the premanufacturing stages, e. g., during the shredding operation whereby the alkali cellulose appears to become more fluffy which enhances subsequent treating operations.

After the addition of the condensate the viscose is otherwise prepared in the normal manner and thereafter extruded through minute spinneret orifices into an acid spinbath to form filaments and the like therefrom. It was determined on examination of the finished rayon that it was more effective than Prestabit Oil in preventing milkiness, even when five times as much Prestibit Oil was used. Furthermore, it was quite effective in inhibiting spinneret incrustation.

Experiments were conducted with respect to solubility and stability of Ethomeen C/25 and S/25 in acid and alkali using 2.0% solutions. The results are tabulated as follows:

Ethomeen	Appearance			
	5% H_2SO_4		6% NaOH	
	Cold	Boiling	Cold	+24 hrs.
C/25	Clear	Clear	Clear	Clear
S/25	Clear	Clear	Clear	Clear

From the above it can be concluded that the compounds in question are adequately soluble and stable in both media.

In conducting several comparative tests for milkiness reduction, Ethomeen C/25 and S/25 were added to the viscose in 0.12% and 0.24% quantities based on the weight of cellulose. These viscoses were spun in the normal manner at the normal maturity of 9.8 and at a higher maturity of 11 to 11.5. On examination of the finished rayon it compared favorably with, or was even superior in appearance to, rayon containing Prestabit Oil.

When the Ethomeens were added to the viscose in the above mentioned quantities and the viscose was spun in an acid spinbath containing no cation active material, it was determined that the pressure build-up behind the spinnerets was reduced markedly, indicating excellent inhibition of spinneret incrustation. This was confirmed by subsequent microscopic examination of the spinnerets.

It can be seen that the use of small amounts of long chain aliphatic tertiary amine-ethylene oxide condensates in accordance with this invention produces several important advantages in the manufacture of viscose rayon. The invention is therefore not to be limited in any manner other than by the scope defined in the appended claims.

What is claimed is:

1. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices into an acid spinbath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a long chain aliphatic tertiary amine-alkylene oxide condensate formed from the reaction of a long chain aliphatic primary amine with an alkylene oxide whereby milkiness in the filaments and incrustation of the orifices are inhibited.

2. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices into an acid spinbath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a small amount of a long chain aliphatic tertiary amine-alkylene oxide condensate formed from the reaction of a long chain aliphatic primary amine with an alkylene oxide whereby milkiness in the filaments and incrustation of the orifices are inhibited.

3. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices into an acid spinbath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of 0.12% to 0.24% based on the weight of the cellulose of a long chain aliphatic tertiary amine-alkylene oxide condensate formed from the reaction of a long chain aliphatic primary amine with an alkylene oxide whereby milkiness in the filaments and incrustation of the orifices are inhibited.

4. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices into an acid spinbath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a long chain aliphatic tertiary amine-ethylene oxide condensate formed from the reaction of a long chain aliphatic primary amine with ethylene oxide whereby milkiness in the filaments and incrustation of the orifices are inhibited.

5. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices into an acid spinbath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a small amount of a long chain aliphatic tertiary amine-ethylene oxide condensate formed from the reaction of a long chain aliphatic primary amine with ethylene oxide whereby milkiness in the filaments and incrustation of the orifices are inhibited.

6. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices into an acid spinbath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of 0.12% to 0.24% based on the weight of the cel-

lulose of a long chain aliphatic tertiary amine-ethylene oxide condensate formed from the reaction of a long chain aliphatic primary amine with ethylene oxide whereby milkiness in the filaments and incrustation of the orifices are inhibited.

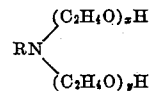
7. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices into an acid spinbath to form filaments and the like therefrom, the step which comprises adding a small amount of a long chain aliphatic tertiary amine-ethylene oxide condensate to the viscose solution and spinning the same into filaments whereby milkiness in the filaments and incrustation of the orifices are inhibited

8. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices into an acid spinbath to form filaments and the like therefrom, the step which comprises adding 0.12% to 0.24% based on the weight of the cellulose of a long chain aliphatic tertiary amine-ethylene oxide condensate to the viscose solution and spinning the same into filaments whereby milkiness in the filaments and incrustation of the orifices are inhibited.

9. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices into an acid spinbath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a long chain aliphatic tertiary amine-ethylene oxide condensate formed from the reaction of a long chain aliphatic primary amine with about 15 moles of ethylene oxide whereby milkiness in the filaments and incrustation of the orifices are inhibited.

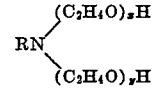
10. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices into an acid spinbath to form filaments and the like therefrom, the step which comprises spinning the viscose filaments in the presence of

a soluble, stable cation active compound having the following formula:



in which R represents an aliphatic chain having at least 8 carbon atoms therein and $x+y$ equals the total number of moles of ethylene oxide at least 5, fortuitously separated into two chains to form the tertiary amine whereby milkiness in the filaments and incrustation of the orifices are inhibited.

11. In the manufacture of viscose yarn wherein a viscose solution is extruded through minute orifices into an acid spinbath to form filament and the like therefrom, the step which comprises spinning the viscose filaments in the presence of a soluble, stable cation active compound having the following formula:



in which R represents an aliphatic chain having at least 8 carbon atoms therein and $x+y$ equals about 15 moles of ethylene oxide, fortuitously separated into two chains to form the tertiary amine whereby milkiness in the filaments and incrustation of the orifices are inhibited.

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The following references are of record in the file of this patent:

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