This invention relates to the manufacture of regenerated cellulose fibres and is particularly concerned with improving the acid dyeing characteristics of such fibres made from viscose.

The cellulose molecule contains no basic groups and, because of this, regenerated cellulose fibres are not dyeable with acid dyes. The acid dyes are a large class developed for wooll-dyeing and comprise a wide range of clear colours which are not as expensive as some of the direct dyes normally used in dyeing cellulose. It would be advantageous to modify cellulose so that regenerated cellulose fibre could be dyed substantially with acid dyes and, indeed, the viscose industry has long sought to do this.

Some of the properties sought after in a dyeing process and in the dyed fibre are, good levelling properties of the dye between the dye sites on the fibre, good wash fastness, good light fastness, a fast rate of exhaustion of the dyebath and a high extent of exhaustion of the dyebath.

It is also desirable that the dye sites in a modified cellulose should be chemically bonded to the cellulose molecule.

We have now found that regenerated cellulose fibres containing aminoethyl ethers of cellulose, for example aminoethyl cellulose and diethylaminoethyl cellulose have very good acid dyeing characteristics. Accordingly the invention comprises a regenerated cellulose fibre containing the aminoethyl and diethylaminoethyl ethers of cellulose.

A regenerated cellulose fibre containing aminoethyl cellulose as the sole amino compound and dyed with acid dye has good fastness to light, but, in general, the fibre does not give a good rate, or extent, of exhaustion of an acid dyebath. A regenerated cellulose fibre containing diethylaminoethyl cellulose as the sole amino compound, results in the fibre giving a good rate and extent of exhaustion of an acid dyebath but the resulting dyed fibre generally tends to have a poorer fastness to light than one containing aminoethyl cellulose. However by using both aminoethyl cellulose and diethylaminoethyl cellulose together, a fibre having good exhaustion of the dyebath and good fastness to light can be obtained.

The aminoethyl ethers of cellulose may be incorporated, or formed in situ, in the cellulose at any suitable stage in the production of the fibre up to the finishing of the fibre.

As applied to the viscose process aminoethyl cellulose and diethylaminoethyl cellulose may be made beforehand and can then be either xanthated and introduced into ordinary viscose, or mixed with the cellulose or alkali cellulose before xanthation. Alternatively, reagents which react with cellulose to form the ether in situ may be added at suitable stages in production. Examples of such reagents are 2-aminoethyl sulphuric acid, 2-chloroethanolamine and the corresponding diethyl compounds.

There are two commercially applied methods of reacting alkali cellulose and carbon disulphide to form sodium cellulose xanthate. In the first, alkali cellulose is dispersed in an inert polar organic liquid, for example acetone, before the carbon disulphide is added, whereas in the second method no such dispersing liquid is used.

There is no difficulty in incorporating up to 5 percent by weight, based on the total weight of the cellulose, of aminoethyl cellulose having a nitrogen content of between 1 and 1.3 percent, in the cellulose used to make a spinning viscose by either of these methods. However only by the first of the two methods is it possible to make a spinning viscose from a cellulose largely or completely modified to aminoethyl cellulose. There is a limit of substantially 5 percent on the weight of aminoethyl cellulose containing between 1 and 1.3 percent of nitrogen which can be incorporated as above in the cellulose, to make a spinning viscose using the second method, since larger quantities than this give a largely insolubilised xanthate, due to cross-linking, from which a viscose suitable for spinning cannot be made. The regenerated cellulose fibre, therefore, preferably contains up to 0.065 percent of nitrogen embodied in amino-ethyl groups.

Diethylaminoethyl cellulose containing between 1 and 1.3 percent of nitrogen, when used in viscose, is preferably present to the extent of 10 to 12 percent by weight based on the total weight of the cellulose, so that the nitrogen content derived from the diethylaminoethyl ether is between 0.1 and 0.15 percent of the total cellulose. Less than this percentage may be used but a lower exhaustion rate of an acid dyebath may then result when the fibres are dyed. Larger quantities may also be usefully employed, but such larger amounts are not recommended as they tend to lower the tenacity of the fibres.

The total nitrogen content of the regenerated fibres derived solely from the amino ethers, is preferably at least 0.15. I have also found that in some cases, notably with acid levelling and acid aggregated type dyes, the fastness to light of dyed regenerated cellulose yarns may be further improved by treating the yarns, before dyeing, with a cross-linking agent.

The following example illustrates one way in which the invention may be carried out:

Example

760 ml of 18.5 percent aqueous caustic soda, 240 ml of water and 2 ml of a wetting agent were mixed together and cooled to 0° C. A powdered mixture of 33 g of aminoethyl cellulose (base capacity 1 meq./g) and 66 g of diethylaminoethyl cellulose (base capacity 1 meq.) were then mixed in and the mixture allowed to stand for 2 hours at 0° C. After this time, 400 g of water and 20 g of acetone were added and the mixture was stirred vigorously and transferred to a water-cooled Pfeiderer mixer where 50 ml of carbon disulphide were added at 18° C. After 2 hours, a further 40 ml of carbon disulphide were added and stirring continued for another 2 hours. 407 g of the resultant viscose were made up to 2 kg, with ordinary viscose to give a product containing 10 percent of diethylaminoethyl cellulose and 5 percent of aminoethyl cellulose, both based on the weight of the cellulose. The combined viscose was spun at a salt figure of 6.2 on a staple fibre machine, the fibres being desulphurised and washed in the usual manner.

The product had an imbition of 100 percent, high resilience, wholly handle, a pronounced wool dyeing affinity for acid-levelling, acid-aggregated and premattalised wool dyes and good light fastness properties when dyed with representative members of these classes of dyestuff. In addition, the high wool dyeing affinity was not significantly reduced by prolonged hot wet treatment in acid media.

What I claim is:

1. Regenerated cellulose fibres containing, uniformly physically dispersed therein, an aminoethyl and a diethylaminoethyl ether of cellulose in proportions such as to
give a total nitrogen content of between about 0.165 and about 0.215 percent by weight of the fibre.

2. Regenerated cellulose fibres as claimed in claim 1 containing at least 0.165 percent by weight of nitrogen derived from the amino ethers.

3. Regenerated cellulose fibres as claimed in claim 1 containing up to 0.065 percent by weight of nitrogen derived from the aminoethyl ether of cellulose component.

4. Regenerated cellulose fibres as claimed in claim 1 containing between 0.10 and 0.15 percent by weight of nitrogen derived from the diethylaminoethyl component.

5. A viscose process for making acid dyeable fibres of regenerated cellulose comprising incorporating a sodium xanthate solution of an aminoethyl ether of cellulose and of a diethylaminoethyl ether of cellulose in a viscose in proportions such as to give a total nitrogen content of between about 0.165 and about 0.215 percent by weight of cellulose.

6. A viscose process for making acid dyeable fibres of regenerated cellulose comprising forming a mixture of aminoethyl and diethylaminoethyl ethers of cellulose with cellulose capable of being xanthated or alkali cellulose in proportions such as to give a total nitrogen content of between about 0.165 and about 0.215 percent by weight of cellulose, and xanthating the mixture.

References Cited by the Examiner
UNITED STATES PATENTS
2,656,241 10/1953 Drake et al. 260—232

ALEXANDER H. BRODMERKEL, Primary Examiner.
L. B. HAYES, Assistant Examiner.