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MANUFACTURE OF CELLULOSE DERIVATIVES HAVING AFFINITY FOR ACID DYESTUFFS

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This invention relates to improvements in the manufacture of organic derivatives of cellulose and of artificial filaments, foils and other articles therefrom.

It has been found that organic derivatives of cellulose which have a very good affinity for acid dyestuffs can be produced by esterifying cellulose with a mixture of chloracetic acid or other halogenated organic acid and acetic anhydride or other organic acid anhydride, so as to produce a cellulose derivative containing both a halogenated ester radicle and another ester radicle, and treating the mixed ester with pyridine or other organic base.

The above invention is of particular importance for the production of filaments, foils and other articles having a good affinity for acid dyestuffs. They may be obtained either by shaping the mixed ester obtained in the first stage and treating the shaped articles with the organic base or by shaping the ester produced after the treatment with the organic base. Articles produced by either of the above methods have a good affinity for acid dyestuffs, even when they have only a low nitrogen content, e. g. .5 to 1%. In most cases the fastness of the colour produced on the dyed articles is greater than the fastness of the corresponding dyestuff on wool. Further, the fastness of the colours to water bleeding and to light is in general very good.

In order to obtain cellulose esters having a high affinity for acid dyes after treatment with the organic base the mixed ester treated should contain not less than 5% of chloracetyl radicle, calculated as chloracetic acid, or an equivalent quantity of halogen if present in another halogenated acid radicle.

The esterifying agents which it is preferred to employ according to the present invention are acetic anhydride and chloracetic acid. The process may be carried out as follows:

100 parts by weight of cotton linters are pretreated with formic acid and are then esterified with a mixture containing about 300–350 parts of acetic anhydride, 50 parts of acetic acid, 250 parts of mono-chloracetic acid and 20 parts of sulphuric acid. The primary ester obtained is ripened in the ordinary way to produce a secondary ester containing 2 to 2.3 acetyl groups and about .2 chloracetyl groups per $C_6H_{10}O_5$ molecule. This is dissolved in acetone and spun into filaments by the ordinary dry spinning process. The filaments obtained are treated in hank form for about 5–7 hours with a 2–4% solution of pyridine in toluene, the temperature of the medium being about 80–100° C. and the volume ratio being 40:1. The yarn is then centrifuged to remove as much of the treatment medium as possible and finally dried at about 60° C.

Instead of treating the yarn with pyridine, the ripened ester in powder form may be treated in the same way with a 2–4% solution of pyridine in toluene, then filtered off, dried and dry spun into filaments from solution in a mixture of 90% methylene chloride and 10% methylated spirit.

The yarn obtained by either of these methods has a good affinity for acid dyestuffs. Dyeing may be effected with a 30:1 aqueous bath at 40° C. the temperature being raised slowly to 80° C. and maintained at this temperature for about an hour. Among acid dyestuffs which may be employed may be mentioned Orange II (Colour Index No. 151) Guinea Fast Red BL (I. G. Wool Dyeing Manual, page 38) Solway Ultra Blue B (Journal of the Society of Dyers and Colourists, 1936, page 238) Solway Green GS, Alizarine Cyanine Green G (Colour Index No. 1078) and Azo Acid Black 3BL (Colour Index, page 342). The yarn is dyed good full shades with all of these dyestuffs, whereas ordinary cellulose acetate yarn is only slightly tinted by Orange II and Solway Ultra Blue B and is not coloured at all by the other three dyestuffs. The yarn can also be dyed with the ordinary water-insoluble cellulose acetate dyestuffs and has a certain degree of affinity for direct cotton colours.

It is important to avoid subjecting the yarns or other materials to treatment with alkaline media such as soap solutions prior to dyeing, since if this is done the affinity for acid dyestuffs may be considerably reduced or even completely destroyed.

Other esterifying agents may be employed according to the present invention instead of acetic anhydride and chloracetic acid, particularly lower fatty acid anhydrides such as propionic and butyric anhydrides and halogen substituted lower fatty acids e. g. chlorpropionic and chlorbutyric acids. Other organic bases which may be employed include di- and tri-ethylamines, piperidine and quinoline.

Yarns produced according to the present invention may be associated, either before or after treatment with the organic base, with ordinary cellulose acetate yarns or with wool, cotton or regenerated cellulose yarns to produce doubled yarns or fabrics which can be dyed with suitable dyestuffs to produce solid, resist or cross-dyed effects according to the dyestuffs employed and the nature of the components present.

Having described our invention, what we desire to secure by Letters Patent is:

1. Process for the production of cellulose derivatives having an affinity for acid dyestuffs, which comprises pretreating 100 parts by weight of cotton linters with formic acid, esterifying said pretreated cellulose with a mixture containing about 300 to 350 parts by weight of acetic anhydride, 50 parts by weight of acetic acid, 250 parts by weight of mono-chloracetic acid, and 20 parts by weight of sulfuric acid, ripening the mixed ester obtained, and treating the ripened mixed ester with pyridine.

2. Process for the production of cellulose derivatives having an affinity for acid dyestuffs, which comprises pretreating 100 parts by weight of cotton linters with formic acid, esterifying said pretreated cellulose with a mixture containing about 300 to 350 parts by weight of acetic anhydride, 50 parts by weight of acetic acid, 250 parts by weight of mono-chloracetic acid, and 20 parts by weight of sulfuric acid, ripening the mixed ester obtained, and treating the ripened mixed ester in powder form with a 2 to 4% solution of pyridine in toluene.

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