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PAD-STEAM DYEING OF POLYACRYLONITRILE TOWS PADDED IN NIP OF SQUEEZE ROLLERS

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2 Claims. (Cl. 8—55)

It is known to unite the continuous synthetic filaments issuing from the spinnerets to form a so-called spun tow.

Hitherto spun tows of polyacrylonitrile have been dyed in steel baskets or in the form of so-called windings which are mounted on spindles. In either case the method used is relatively troublesome and not very efficient (cf. SVF-Fachorgan 12, page 358 [1957]).

The present invention is based on the observation that spun tows of polyacrylonitrile or of copolymers of which acrylonitrile is the main component can be dyed in a continuous manner by impregnating the material with an aqueous solution or dispersion of a dyestuff, and treating the impregnated material with steam to fix the dyestuff thereon.

The impregnation with the dyestuff is advantageously carried out by padding, that is to say, by passing the spun tow to be dyed through a padding bath and then squeezing the material in the usual manner, advantageously until the impregnated material retains 50 to 130 percent of its initial weight of dyestuff solution. The dyestuff used may be the same as those used for the known processes for dyeing polyacrylonitrile fibers, for example, dispersion dyestuffs of the type which are used for dyeing cellulose esters or basic dyestuffs, such as triphenylmethane dyestuffs, oxazine dyestuffs, methine dyestuffs, azo-dyestuffs or anthraquinone dyestuffs, which contain tertiary or quaternary amino groups. In general, it is of advantage to use a dyebath having a concentration of dyestuffs of 10 to 50 grams per liter of dye liquor and to maintain a weakly acid reaction in the dyebath, advantageously by adding acetic acid. The temperature of the padding bath is not in itself critical, but, as relatively concentrated dyestuff solutions are used, it is advantageously at least 40° C. to precent any precipitation of the dyestuff.

The fixation of the dyestuff on the material impregnated with the dyestuff solution is carried out by steaming, preferably at a temperature above 100° C.

It is especially advantageous to carry out this treatment in a pressure chamber of the kind described in British specification No. 823,022, issued August 14, 1959, to Konrad Peter Aktiengesellschaft, Liestal, Switzerland, in which the material is treated under super-atmospheric pressure, for example, with saturated steam. To enable this treatment to be carried out in a continuous manner, and at the same time to maintain the superatmospheric pressure in the chamber, liquid-free pressure-tight packing devices are provided at the points of entry and exit of the tow into and from the chamber. These packing devices each comprise two rollers rotatably mounted on the chamber housing, between which the material is passed under resilient clamping pressure. Particularly good results are obtained with pressure chambers in

which the steam packings are designed as two pairs of rollers made of different materials, the spun tow coming into contact only with one pair of rollers. As the speed of fixation of the dyestuff depends on the temperature in the interior of the pressure chamber, it is of advantage to work at a temperature above 100° C., for example, within the range of 100 to 150° C. With most dyestuffs fixation is achieved at a temperature of 105 to 110° C. or in some cases up to 120° C., so rapidly that the residence time of the spun tow in the pressure chamber is only about 1 to 2 minutes.

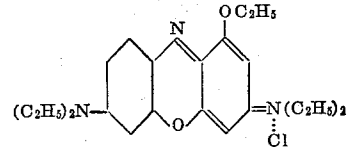
The process of this invention yields strong and pure dyeings. It is surprising that even a thick spun tow is dyed uniformly throughout, notwithstanding the short duration of its passage through the dyebath.

If desired, the dyed spun tow may be subjected to one or more after-treatments. To remove any unfixed dyestuff from the tow, it is of advantage to soap the dyed tow, that is to say, to treat it with a solution which contains soap or a non-ionic synthetic detergent and has a temperature of 60° C. to 90° C.

The following examples illustrate the invention, the parts and percentages being by weight:

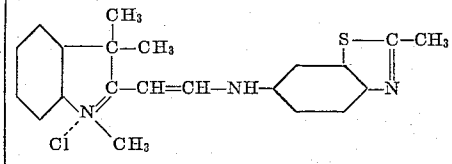
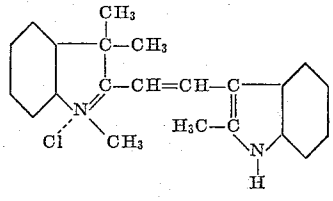
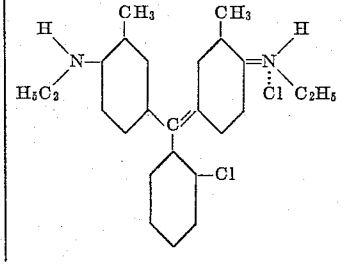
Example 1

30 parts of the dyestuff of the formula



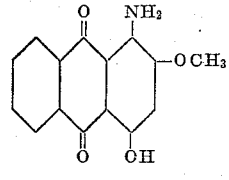
are pasted with 30 parts of acetic acid of 40% strength, and the paste is then made up to 1000 parts with boiling water. A spun tow of polyacrylonitrile (registered trademark Orlon) is padded with the dyestuff solution and then squeezed until its increase in weight is 100%. The tow is then passed through a pressure chamber, such as is described in the above mentioned British specification No. 823,022, which comprises at the inlet and outlet of the textile web a pair of rollers rotatably mounted on the chamber housing, between which two pairs of rollers the material passes under resilient clamping pressure, and which rollers are sealed, on the one hand, by means of plates located on the housing and co-operating in front with the ends of the rollers and, on the other, by means of elements fixed to the housing and pressing on the inside against the roller shells. The speed of travel is so controlled that an optimum fixation of the dyestuff on the fiber is achieved. In the present instance the passage of the spun tow takes about one minute. The spun tow leaving the pressure chamber through the outlet rollers is dyed a strong pure blue tint of remarkably level character. To remove any unfixed dyestuff therefrom, the tow is passed through a solution maintained at 90° C. and containing in 1000 parts of water 1 part of an aqueous solution of 30% strength of the addition compound of 15 molecular proportions of ethylene oxide and 1 molecular proportion of cetyl alcohol.

In the following table are given further dyestuffs which dye spun tows of polyacrylonitrile by the process described in the preceding paragraph the tints given in column II of the table.

	I	II
2		yellow.
3		orange.
4		blue.

Example 2

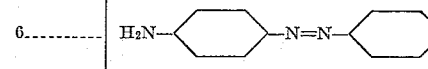
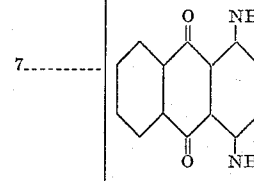
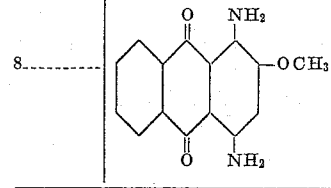
20 parts of the dyestuff of the formula



are ground with 40 parts of an aqueous solution of 50% strength of sulfite cellulose waste liquor and then dried. This dyestuff preparation is dispersed in water with 10 parts of the sodium salt of N-benzyl-μ-heptadecyl benzimidazole disulfonic acid and 6 parts of crystal gum as thickener, and the whole is made up with water to 1000 parts. An Orlon spun tow is dyed with this solution by the method described in Example 1, and is then soaped. A pure pink dyeing is obtained.

In the following table are given further dyestuffs which, when dispersed as described in the preceding paragraph,

dye spun tows of polyacrylonitrile by the method described in Example 1 the tints given in column II:

	I	II
6		orange.
7		violet.
8		pink.

What is claimed is:

1. A process for dyeing a tow of a member selected from the group consisting of polyacrylonitrile and a copolymer of which acrylonitrile is the main component, which consists essentially of moving the tow in a continuous manner and in immediate succession

(*a*) through a bath in the nip of, and directly through, a pair of squeezing rollers, which bath is a member selected from the group consisting of an aqueous solution of a water-soluble basic dyestuff and an aqueous dispersion of a dispersion dyestuff;

(*b*) through a pair of squeezing, pressure sealing rollers,

(*c*) through substantially saturated steam under superatmospheric pressure at about 105° to 140° C. for a period of about one to two minutes, and

(*d*) between a second pair of squeezing, pressure sealing rollers, both pairs of pressure-sealing rollers sealing the substantially saturated steam in a chamber through which said tow is moved, whereby the tow is uniformly dyed throughout and the dyestuff is fixed thereon.

2. A process as claimed in claim 1, wherein the steam treatment is carried out at a temperature within the range of 105° C. to 120° C.

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