

[54] **PROCESS FOR RESERVING TEXTILES OF NATURAL POLYAMIDE FIBRES AND OF SYNTHETIC FIBRES DYEABLE WITH ACID DYES**

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[58] Field of Search 8/17, 66, 15, 1.2, 8/163, 115.5

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[57] **ABSTRACT**

A process for the reservation of natural polyamide fibres against anionic dyes and acid dyeable synthetic fibres, which process consists of applying a solution or dispersion of one or more colourless, fibre-reactive compounds to areas of a material (composed by such fibres) that are to be reserved, and fixing the compound or compounds prior to coloration of the material.

6 Claims, No Drawings

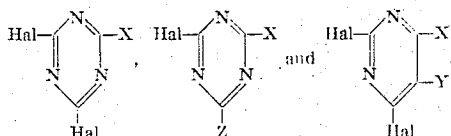
PROCESS FOR RESERVING TEXTILES OF NATURAL POLYAMIDE FIBRES AND OF SYNTHETIC FIBRES DYEABLE WITH ACID DYES

This invention relates to a process for the local reservation against anionic dyes of textiles made of natural polyamide fibres and of textiles of synthetic fibres dyeable with acid dyes, which process consists in impregnation of the fibre with colourless fibre-reactive compounds and fixation of the impregnation.

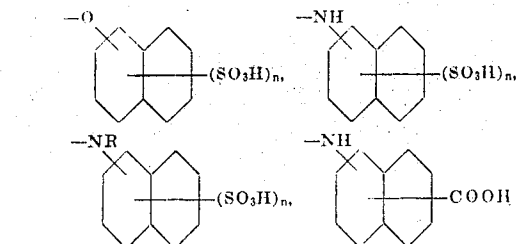
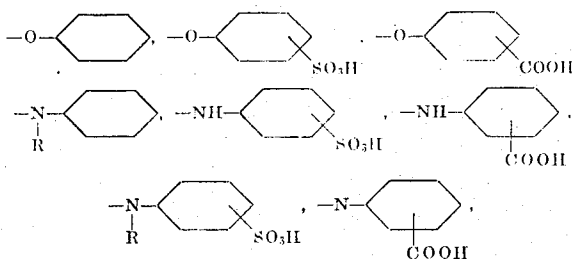
It has been found that natural polyamide fibres and synthetic fibres dyeable with acid dyes (c.f. Colour Index, Second Edition, 1956, Volume I, pages 1,001 - 1,404) can be locally reserved against anionic (acid) dyes by impregnating the areas intended for reservation with a liquor or a paste containing one or more colourless fibre-reactive compounds and then fixing the impregnation.

The term "natural polyamide fibres" refers in particular to filaments, yarns, woven fabrics or knitted goods of wool or silk. Under "synthetic fibres dyeable with acid dyes" are understood the aforementioned textile forms of synthetic polyamide fibres, e.g. nylon 6, 7, 11, 66, 76, 226, 610 and 6/66, of basic modified polypropylene and of basic modified polyacrylonitrile and acrylonitrile copolymer fibres.

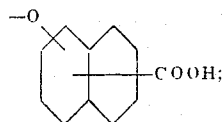
The colourless fibre-reactive compounds employed for impregnation contain a colourless organic radical of any desired type which may bear substituents and/or hetero atoms, is preferably not appreciably volatile at 100°C, and has attached to it a group which reacts readily with a nitrogen atom of the textile material for reservation. Suitable compounds of this type include heterocyclic compounds having 1 or 2 exchangeable halogen atoms, for example those of the formulae



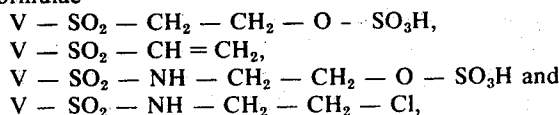
where Hal stands for F, Cl or Br, X for a group of one of the formulae



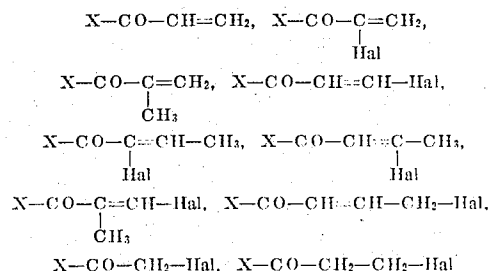
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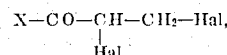
Y for H, F or Cl; Z for an unsubstituted amino group, an amino group substituted by one or two alkyl groups with one to four carbon atoms, or an alkoxy group with one to four carbon atoms; n for 0, 1, 2 or 3; and R for an alkyl group having one to four carbon atoms; and where the $-\text{SO}_3\text{H}$ and $-\text{COOH}$ groups are attached to the aromatic nucleus at positions other than those ortho to the oxygen or imino bridge member and the aromatic nuclei may be substituted by, for example, halogen atoms or nitro, cyano or alkyl groups having one to four carbon atoms. Other suitable compounds are those which contain sulphonyl groups and double bonds capable of addition or groupings capable of forming double bonds; examples are compounds of the formulae



where V represents a phenyl radical which may be substituted by, e.g., $-\text{Cl}$ or $-\text{CH}_3$, and compounds containing aliphatic groups and a double bond capable of addition or an exchangeable halogen atom, e.g. those of the formulae



and



where X has one of the aforesaid meanings and Hal represents a chlorine or bromine atom.

For the process of this invention compounds containing one carboxylic or sulphonc acid group are preferred. Compounds with a pyrimidine nucleus are very stable to hydrolysis in neutral and alkaline medium. Cyanuric acid derivatives react extremely rapidly, while trifluoropyrimidyl derivatives are particularly suitable for the reservation of natural polyamide fibres. In selecting suitable colourless fibre-reactive compounds, the textile colourist will be guided by these known criteria.

The areas of the textile substrate treated with these colourless fibre-reactive compounds, besides being reserved against anionic dyes, generally exhibit increased dyeability with basic dyes.

If the compounds are applied by a printing process, the known method of print paste preparation for dyes is employed. Water-insoluble compounds are applied in aqueous dispersion. For this purpose they are preferably ground to an average particle size of about 0.1 to 10 microns or more particularly 0.1 to 5 microns, if necessary in the presence of dispersing agents or fillers. For example, the dry compounds can be ground with a dispersing agent and if required fillers, or alternatively it can be kneaded in paste form with a dispersing agent and subsequently vacuum or jet dried.

It is advisable to add an acid-binding agent, such as sodium or potassium carbonate or bicarbonate, to preparations of compounds containing reactive halogen, so that the hydrogen halide liberated in fixation is immediately neutralized.

One of the commonly used thickening agents is normally included in the padding liquor or printing paste, for example modified or unmodified natural products, such as sodium alginate, British gum, gum Arabic, crystal gum, locust bean gum, gum tragacanth, starch, carboxymethyl-cellulose or hydroxyethyl-cellulose, or synthetic products, such as polyacrylamides or polyvinyl alcohols.

The padding liquor or printing paste may contain the colourless fibre-reactive compounds in amounts of up to about 150 parts per 1,000 parts. Fixation is effected preferably in a moist atmosphere at room temperature for periods of up to about 20 hours, or between 25° and 95°C for 2 hours, or between 95° and 130°C for about 15 minutes.

Polyamide materials treated with the colourless fibre-reactive compounds can be dyed with anionic dyes; the areas of the material treated with the compounds then remain undyed or are dyed to lighter depth than the remaining material, depending on the degree of reservation imparted by the treatment. The best reserve effects are obtained against anionic dyes bearing two or more sulphonate acid groups; in fact 100 percent reservation of synthetic polyamide fibres can be obtained against this type of dye. A step by step decrease in the degree of reservation is obtained with dyes bearing only one sulphonate acid group and with premetallized dyes. This process of reservation has no effect on disperse dyes which give the normal effects obtained with this class, whereas basic dyes dye the reserved areas of polyamide material to greater depth than the unreserved substrate. When fibre-reactive compounds containing one or more acid groups are employed for reservation, the material can be subsequently dyed with anionic dyes and then with basic dyes to produce two-colour effects, since the latter class preferentially dyes the areas reserved against anionic dyes. The reverse procedure is of course practicable, with the basic dyes being applied first to dye the reserved areas, followed by anionic dyes to fill in the remaining material. Dyes of these two classes can be applied from the same bath if desired.

In the following Examples the parts and percentages are by weight and the temperatures in degrees centigrade.

Example 1

Carpet yarn of texturized nylon 66 fibre is locally impregnated with a paste of the following composition at a pressure giving a 100 percent increase over the dry weight on the impregnated areas:

- 10 parts sodium alginate thickening
- 40 parts sodium carbonate, anhydrous
- 840 parts water,
- 9 parts octylphenyl-pettaglycol ether,
- 1 part of modified sodium alkylsulphate
- 40 parts β -naphthoxy-dichlorotriazine dispersed with
- 60 parts sodium dinaphthymethanedisulphonate.

Before application the paste is vigorously stirred until homogeneous. The impregnated yarn is stored for 2 hours at room temperature (20°–25°) and then rinsed with cold water. It is tufted in a jute fabric and the tufted carpet exhaustion dyed with two dye mixtures, one of C.I. 11855 Disperse Yellow 3 and C.I. 17045

Acid Red 37, the other of C.I. 61505 Disperse Blue 3 and C.I. 17045 Acid Red 37. In this way a two-colour dyeing is obtained.

Example 2

- 5 A printing paste of the following composition:
- 30 parts sodium alginate thickening
 - 40 parts sodium bicarbonate, anhydrous
 - 804 parts water,
 - 6 parts modified sodium alkylsulphate
 - 10 30 parts β -naphthoxy-dichlorotriazine dispersed with
 - 90 parts sodium dinaphthylmethanedisulphonate
- is applied to a nylon 66 fabric by the Vigoureux process at a coverage of 50 percent and a pressure giving a 100 percent increase over the dry weight on the printed areas. Prior to application the paste is vigorously stirred until of homogeneous consistency. After printing, the fabric is treated in a saturated steam atmosphere for 10 minutes at 105° to fix the impregnation and then rinsed with cold water.

- 20 The fabric, thus locally preserved against anionic dyes, is cut into five equal parts and these are dyed, respectively, with the following dyes:

- 1% C.I. 17045 Acid Red 37 (2 sulphonate acid groups),
- 25 1% C.I. Acid Red 57 (1 sulphonate acid group),
- 1.5% C.I. Acid Red 216 (premetallized dye),
- 1% C.I. Disperse Red 43 (disperse dye),
- 1% C.I. Basic Red 44 (basic dye).

- The length of fabric dyed with C.I. 17045 Acid Red 37 after rinsing and drying, is left practically undyed on the reserved areas. In the piece dyed with C.I. Acid Red 57 the reserved sites are dyed to light depth, and in the piece dyed with C.I. Acid Red 216 to slightly heavier depth. The cutting dyed with C.I. Disperse Red 43 exhibits no depth difference between the reserved and unreserved areas. In that dyed with C.I. Basic Red 44 the reserved areas are dyed to greater depth than the unreserved.

Example 3

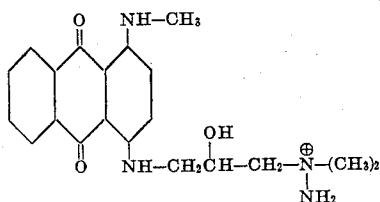
- 40 A printing paste of the following composition:
- 30 parts sodium alginate thickening
 - 40 parts sodium bicarbonate, anhydrous
 - 790 parts water
 - 18 parts octylphenyl-pentaglycol ether
 - 45 2 parts modified sodium alkylsulphate
 - 30 parts β -naphthoxy-dichlorotriazine dispersed with
 - 90 parts dinaphthylmethane disulphonate
- is printed on nylon 6 yarn by the Vigoureux process at a coverage of 25 percent and a pressure giving an increase of 100 percent over the dry weight on the printed areas. Before application the paste is vigorously stirred until homogeneous. The printed yarn is treated in a saturated steam atmosphere for 10 minutes at 105° and then rinsed with cold water. Equal parts of the yarn are exhaustion dyed from one bath with two dye mixtures, one of C.I. 11855 Disperse Yellow 3 and C.I. 61125 Acid Blue 23, the other of C.I. Disperse Blue 2 and C.I. 17045 Acid Red 37. In both lots of yarn the reserved areas are dyed by the disperse dye alone, whilst the unreserved areas are dyed by the disperse and acid dyes. In this way two-colour dyeings are obtained.

Example 4

- A printing paste is prepared with:
- 65 40 parts silicone thickening
 - 20 parts sodium bicarbonate, anhydrous
 - 804 parts water
 - 6 parts modified sodium alkylsulphate

40 parts phenoxy-dichlorotriazine dispersed with
90 parts sodium dinaphthylmethanedisulphonate

After homogenization by vigorous stirring the paste is printed on a nylon 66 fabric at a pressure giving a 100 percent increase over the dry weight on the printed areas. The impregnation is fixed in a saturated steam atmosphere for 5 minutes at 100° and the fabric then rinsed with cold water. It is dyed by the normal exhaustion method with the basic dye of the formula



On the treated areas the fabric is dyed to considerably greater depth than in the remaining substrate.

Example 5

A wool yarn is locally padded with a paste of the following composition at a 100 percent increase over the dry weight on the impregnated areas:

- 10 parts sodium alginate thickening
- 50 parts sodium bicarbonate
- 830 parts water
- 9 parts octylphenyl-pentaglycol ether
- 1 part modified sodium alkylsulphate
- 40 parts β -naphthoxydichlorotriazine, dispersed in
- 60 parts sodium dinaphthylmethanedisulphonate

The paste is homogenized by vigorous stirring before application, and after padding the yarn is stored for 15 minutes at room temperature, then rinsed with cold water and dried. It is woven into fabric and this is exhaustion dyed with Acid Light Scarlet GL C.I. 23905 (Acid Red 145). On the impregnated areas the depth of the dyeing is approximately 10 percent of that on the untreated areas of the fabric.

A similar dyeing is obtained when in place of 50 parts of sodium bicarbonate 50 parts of ammonium bicarbonate are used and the impregnation is fixed in a saturated steam atmosphere for 15 minutes at 100°.

Example 6

A paste is prepared with

- 5 parts sodium alginate thickening
- 30 parts disodium phosphate (anhydrous)
- 2 parts monosodium phosphate (anhydrous)
- 917 parts water
- 8 parts octylphenyl-pentaglycol ether
- 2 parts modified sodium alkylsulphate
- 9 parts 2,4-dichloro-6-phenylamino-1,3,5-triazine, dispersed with
- 27 parts sodium dinaphthylmethanedisulphonate

and homogenized by vigorous stirring. It is printed on carpet yarn of texturized nylon 66 fibre at a 100 percent increase over the dry weight on the printed areas, stored for one day at room temperature (20°-25°) and then rinsed with warm and cold water. The treated yarn is tufted on a jute backing fabric and exhaustion dyed with a mixture of C.I. 11855 Disperse Yellow 3 and C.I. 61125 Acid Blue 23. A two-colour dyeing with sharp colour differentiation is obtained.

Example 7

A printing paste is prepared with the following additions and vigorously stirred until homogeneous:

- 10 parts sodium alginate thickening

- 30 parts anhydrous disodium phosphate
- 2 parts anhydrous monosodium phosphate
- 921 parts water

- 24 parts octylphenyl-pentaglycol ether
- 6 parts modified sodium alkylsulphate
- 7 parts sodium 2,4-dichloro-6-phenylamino-1,3,5-triazine-4'-sulphonate.

The paste is printed on a nylon 6 fabric by the Vigoureux process at a coverage of 25 percent and a pressure giving a 100 percent increase over the dry weight on the printed areas. The fabric is stored for 24 hours at room temperature (20°-25°) and rinsed with cold water. It is then exhaustion dyed with C.I. Acid Red 37, which results in a pale red dyeing on the treated and a dark red dyeing on the untreated areas.

Example 8

Carpet yarn of texturized nylon 66 fibre is impregnated at a 100 percent increase over the dry weight on the impregnated areas with a homogeneously stirred paste of the composition:

- 5 parts sodium alginate thickening
- 30 parts anhydrous disodium phosphate
- 2 parts anhydrous monosodium phosphate
- 933 parts water
- 8 parts octylphenyl-pentaglycol ether
- 2 parts modified sodium alkylsulphate
- 5 parts 2,4-dichloro-6-phenylamino-1,3,5-triazine, dispersed with
- 15 parts sodium dinaphthylmethanedisulphonate.

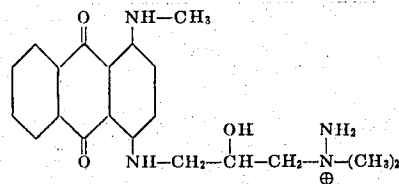
The yarn is stored for 2 hours at 40°, rinsed with warm and cold water, and dyed with C.I. 61505 Disperse Blue 3 and C.I. Acid Red 37 to produce a two-colour effect on the treated and untreated areas respectively.

Example 9

Texturized nylon 6 yarn in package form is locally impregnated with a homogeneously stirred liquor of the following composition at a 100 percent increase over the dry weight on the printed areas:

- 1 part sodium alginate thickening
- 30 parts anhydrous disodium phosphate
- 2 parts anhydrous monosodium phosphate
- 949 parts water
- 8 parts octylphenyl-pentaglycol ether
- 2 parts modified sodium alkylsulphate
- 7 parts sodium 2,4-dichloro-6-phenylamino-1,3,5-triazine-4'-sulphonate.

The yarn is stored for 2 hours at 40°, rinsed with warm and cold water, and exhaustion dyed with C.I. Acid Red 37 and the basic dye of the formula



with the addition of a mixture of carboxymethylated oleyldecaglycol ether and the condensation product of 2-stearyl-3-hydroxy-propyl-diethylenetriamine with 10 moles of ethylene oxide. The impregnated areas are dyed dark blue, the untreated areas red.

Example 10

- A printing paste is prepared with
- 5 parts sodium alginate thickening
- 30 parts anhydrous disodium phosphate

- 2 parts anhydrous monosodium phosphate
933 parts water
8 parts octylphenyl-pentaglycol ether
2 parts modified sodium alkyl sulphate
5 parts 2,4-dichloro-6-phenylamino-1,3,5-triazine 5
dispersed with
15 parts sodium dinaphthylmethanedisulphonate,

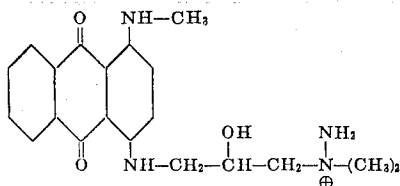
The paste is vigorously stirred until homogeneous and printed locally on nylon 66 yarn at a 100 percent increase over the dry weight on the printed areas. The 10 yarn is then treated for 4 minutes at 98°, rinsed with warm and cold water, and dyed in the normal way with C.I. Acid Red 57. The treated areas are of pale red shade; the untreated, dark red.

Example 11

A paste is prepared with

- 5 parts sodium alginate thickening
30 parts anhydrous disodium phosphate
2 parts anhydrous monosodium phosphate
944 parts water
8 parts octylphenyl-pentaglycol ether
2 parts modified sodium alkylsulphate
7 parts sodium 2,4-dichloro-6-phenylamino-1,3,5-triazine-4'-sulphonate.

The paste is vigorously stirred until homogeneous and printed on nylon 6 yarn at a 100 percent increase over the dry weight on the printed areas. The yarn is treated for 4 minutes at 102° and rinsed with cold water. It is woven into fabric which is exhaustion dyed with a basic blue of the formula



to give a tone-in-tone dyeing of varying strength.

Example 12

A printing paste is made up with

- 5 parts sodium alginate thickening
35 parts anhydrous phosphate buffer mixture giving a pH value of 8
885 parts water
24 parts octylphenylpentaglycol ether
6 parts modified sodium alkylsulphate
45 parts sodium-2,4-dichloro- 6-phenylamino-1,3,5-triazine-4'-sulphonate.

It is vigorously stirred until homogeneous and printed on wool yarn at a 100 percent weight increase on the printed areas. The yarn is stored for 1 hour at 60°, rinsed with warm and cold water, and exhaustion dyed with a mixture of C.I. Acid Blue 23 and C.I. 18835 Acid Yellow 25. A two-colour dyeing with clear depth differentiation is obtained.

Example 13

Unweighted silk yarn is printed as described in Example 12, treated for 10 minutes at 100° in a wet steam atmosphere, and exhaustion dyed with C.I. Acid Red 145. The impregnated fibre sites are dyed to considerably less depth than the remainder of the yarn.

Example 14

A paste of the following composition is prepared and homogenized by powerful stirring:

- 5 parts sodium alginate thickening
30 parts anhydrous disodium phosphate

- 5 parts anhydrous trisodium phosphate
24 parts octylphenyl-pentaglycol ether
6 parts modified sodium alkylsulphate
900 parts water
30 parts sodium 2,4-dichloro-6-phenylamino-1,3,5-triazine-4'-sulphonate.

The paste is locally printed on a polypropylene yarn rendered acid dyeable by chemical modification. The yarn is steamed in wet steam for 15 minutes at 100°, rinsed with warm and cold water, and exhaustion dyed with C.I. Acid Orange 43. The treated areas are dyed to pale depth; the untreated, to heavy depth.

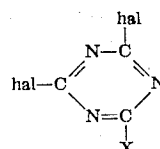
Example 15

A paste of the following composition

- 15 5 parts sodium alginate thickening
30 parts anhydrous disodium phosphate
5 parts anhydrous monosodium phosphate
24 parts octylphenyl-pentaglycol ether
6 parts modified sodium alkylsulphate
20 400 parts water and
30 parts sodium 2,4-dichloro-6-phenylamino-1,3,5-triazine-4'-sulphonate

is homogenized by thorough stirring and printed on yarn of modified acrylic fibre. The yarn is steamed for 15 minutes at 110° in saturated steam and then dyed with C.I. Acid Orange 43. The treated areas are dyed to considerably lighter depth than the untreated.

The following table contains further examples of dyeings with partial reservation on natural or synthetic polyamide fibres, on basic modified polypropylene fibres or on basic modified acrylonitrile polymer fibres. These dyeings are carried out according to the details of Examples 1 to 15 and are characterized in the table by the textile material employed, the substituents hal 35 and X in the reserving agent of the formula



and by the procedure used.

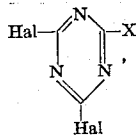
Table

Example No.	Textile material	hal	Substituent X	Procedure according to Example (IV)
50	16 wool yarn	Br	4-sulphophenylamino	12
	17 nylon 66	Cl	3-methylphenylamino	8
	18 nylon 66/6	Cl	4-methylphenoxy	4
	19 nylon 6	Cl	3-sulphophenylamino	9
	20 do.	Cl	4-carboxyphenoxy	9
	21 nylon 610	Cl	4-chlorophenoxy	4
	22 nylon 11	Cl	3-sulphophenylamino	7
55	23 nylon 66	Cl	4-sulphonaphthyl-1-amino	9
	24 do.	Cl	6-sulpho-naphthoxy-2	9
	25 do.	Cl	6,8-disulphonaphthoxy-2	9
	26 do.	Cl	4-sulphophenoxy	9
	27 nylon 6	Cl	3-chlorophenylamino	8
	28 do.	Cl	4-methylphenylamino	8
	29 do.	Cl	naphthoxy-1	3
60	30 do.	Cl	3-carboxyphenylamino	6
	31 do.	Cl	2,5-disulphophenylamino	7
	32 nylon 66	Cl	2,4-disulphophenylamino	7
	33 wool yarn	Cl	4-carboxyphenylamino	12
	34 do.	Cl	4-chlorophenylamino	5
	35 nylon 6	Cl	4-sulphonaphthoxy-1	9
	36 do.	Cl	4,6-disulphonaphthoxy-1	9
65	37 do.	Cl	7-sulphonaphthyl-1-amino	7
	38 do.	Cl	4,8-disulphonaphthyl-2-amino	7
	39 nylon 6	Cl	5,7-disulphonaphthyl-1-amino	7

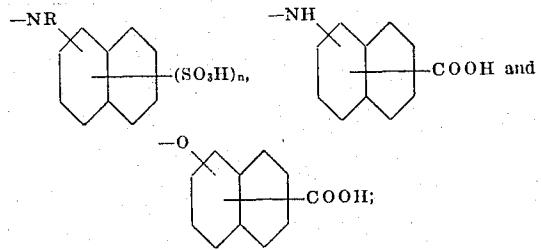
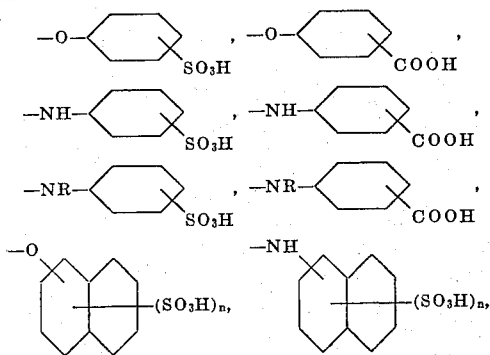
40	nylon 6/66	Cl	6-sulphonaphthyl-2-amino	7
41	nylon 66	Cl	N-methyl-N-phenylamino	8
42	nylon 6	Cl	N-ethyl-N-phenylamino	10
43	do.	Cl	N-methyl-N-(6-sulphonaphthyl-2)-amino	11
44	nylon 66	Cl	N-ethyl-N-(3-methylphenyl)-amino	10
45	do.	Cl	N-methyl-N-(4-sulphophenyl)-amino	11
46	nylon 6	Br	4-sulphophenylamino	9
47	do.	F	do.	9
48	do.	F	Phenylamino	8
49	wool yarn	F	do.	5
50	nylon 6	Cl	4,6,8-trisulphonaphthyl-2-amino	7
51	do.	Cl	3,6,8-trisulphonaphthyl-1-amino	7
52	do.	Cl	3,6,8-trisulphonaphthoxy-1	7
53	PP*	Cl	4-sulphonaphthyl-1-amino	14
54	PAN*	Cl	3-sulphophenylamino	15
55	nylon 66	Cl	3,5-disulphophenylamino	9

PP* = basic modified polypropylene yarn
PAN* = modified polyacrylonitrile yarn

Having thus disclosed the invention what we claim is:
1. In a process for local reservation against anionic dye of synthetic polyamide fibre dyeable with acid dye, the improvement consisting essentially of (a) padding or printing a solution of a compound to an area of the fibre which is to be reserved and (b) fixing the compound on the fibre prior to coloring said fibre; the compound being of the formula



wherein Hal is fluoro, chloro or bromo;
X is a group of one of the formulae



R is alkyl having from one to four carbon atoms, inclusive;
n is 1, 2 or 3; and
wherein each HO₃S— and each HOOC—group is bound to an aromatic nucleus at a position other than one ortho to an oxygen or imino bridge member; and each aromatic nucleus is either further substituted or further unsubstituted, any substituent on a further substituted aromatic nucleus being a member selected from the group consisting of halo, nitro, cyano and alkyl having from one to four carbon atoms.

2. A process according to claim 1 wherein the synthetic polyamide fibre is a nylon fibre.

3. A process, according to claim 1, wherein the compound is fixed on the fiber by reacting the fibre-reactive compound with a nitrogen atom of the fiber.

4. A process according to claim 3 wherein fixing comprises reacting the fibre-reactive compound with a nitrogen atom of the fibre, whereupon hydrogen halide is liberated, said fibre-reactive compound being sodium 2,4-dichloro-6-phenylamino-1,3,5-triazine-4'-sulfonate.

5. A process according to claim 3 wherein fixing comprises reacting the fibre-reactive compound with a nitrogen atom of the fibre, whereupon hydrogen halide is liberated, said fibre-reactive compound being sodium 2,4-dichloro-6-phenylamino-1,3,5-triazine-2',5'-disulfonic acid.

6. A process according to claim 1 wherein the fibre is in textile form.

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