

PATENT SPECIFICATION

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(54) COLORATION PROCESS

(71) We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, of Imperial Chemical House, Millbank, S.W.1., a British Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a coloration process and more particularly to a process for coloring synthetic textile materials.

It is well known to color synthetic textile materials or unions containing synthetic textile materials, by applying to the textile material an aqueous dispersion of a dyestuff, drying the textile material and then subjecting it to a dry heat treatment involving baking or steaming using superheated steam. During this dry heat treatment transfer of the dyestuff takes place within the synthetic textile material, or, in the case of unions of synthetic textile materials with protein or cellulose textile materials, transfer of the dyestuff takes place from the protein or cellulose (which are preferentially colored in the initial stages) to the synthetic textile materials. However, during the past few years it has been found necessary to produce dyestuffs for synthetic textile materials which have much higher fastness to heat treatments such as are used in pleating operations, and it has been found that such dyestuffs are not so readily transferred so that it has become increasingly difficult to obtain satisfactory coloration of the synthetic textile materials.

It has now been found that this difficulty can be overcome by carrying out the whole or part of the dry heat treatment at pressures less than atmosphere pressure.

According to the invention there is provided a process for coloring synthetic textile materials or unions containing synthetic textile materials which comprises applying a water-insoluble dyestuff free from fibre-reactive groups to the textile material and then subjecting the textile material to a dry heat

treatment at a pressure less than atmospheric pressure.

The dyestuff can be applied to the textile material in the form of an aqueous dispersion, the textile material is dried, and then subjected to the dry heat treatment which comprises baking the textile material at temperatures above 100°C, preferably at temperatures between 110° and 240°C, or subjecting the dried textile material to the action of superheated steam, preferably superheated steam having a temperature of between 110° and 240°C. The dry heat treatment is carried out at pressures below atmospheric pressure and preferably at pressures of between 0.1 and 100 mms. of mercury. If desired, part of the dry heat treatment can be carried out at atmospheric pressure. It is generally found that the dry heat treatment need only be carried out for short periods, and in particular it is found that times of between 30 seconds and 10 minutes are adequate to obtain satisfactory results. If desired the baking treatment can be carried out by means of microwave heating or infra red heating.

The dyestuff, or mixtures of dyestuffs, can be applied to the textile material by dyeing, padding or printing processes using either a dye liquor, padding liquor or print paste containing the dyestuff in the form of an aqueous dispersion. The said liquors or pastes can contain the conventional additives, such as anionic, non-ionic and/or cationic dispersing agents, urea, sodium *m* - nitrobenzene sulphonate, thickening agents such as alginates or gums, or organic liquids such as benzyl alcohol. When a union of a synthetic textile material and a non-synthetic textile material is being colored by the process of the invention then the said liquor or paste can also contain a dyestuff, usually a water-soluble dyestuff, for the non-synthetic textile material.

The term "free from fibre-reactive groups" denotes that the dyestuffs do not contain any groups, such as β - chloroethylsulphonamide groups, which will chemically combine with

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SPECIFICATION AMENDED - SEE ATTACHED SLIP

amino groups present in synthetic polyamide textile materials.

As examples of the water-insoluble dyestuffs which can be used in the process of the invention there may be mentioned Disperse Dyestuffs such as are described in, for example, pages 1665 to 1742 of the second edition of the Colour Index which was published in 1956, or in British Patent Specifications Nos. 806271, 835819, 847175, 852396, 852493, 859899, 865328, 872204, 894012, 908656, 909843, 910306, 913856, 919424, 944513, 944722, 953887, 959816, 960235, 961412, 988629, 976218, 993162, 998858, 1012238, 1012239, 1016246, 1016651, 1037649, 1020303, 1047089, 1048250, 1048882, 1049039, 1053830, 1053997 and 1056354. There can also be used Vat Dyestuffs such as are described in, for example, pages 2419 to 2564 of the second Edition of the Colour Index.

Alternatively the process of the invention can be carried out by a dye transfer technique, in which a print paste containing the water-insoluble dyestuff is printed onto a cellulose material, such as paper, which is optionally dried, and which is then placed in contact with the synthetic textile material, and the whole subjected to a dry heat treatment at less than atmospheric pressure. This process is advantageously carried out in a continuous manner, in which case the cellulose material and the synthetic textile material are passed together between heated rollers, the pressure round the rollers being maintained at less than atmospheric pressure.

The print pastes used in this process can be either water-based inks or oil-based inks, that is to say the dyestuffs and the other ingredients are dispersed or dissolved in water or in an organic solvent medium, for example in a medium comprising a solution of zinc rosinate in toluene or a solution in ethanol of a partial ester of pentaerythritol with a mixture or rosin and fumaric acid.

At the conclusion of the process the coloured textile material is preferably rinsed in water and/or given a treatment in an aqueous solution of soap or a synthetic detergent before being dried. In the case of aromatic polyester textile materials it is also preferred to subject the coloured textile material to a treatment in an alkaline aqueous solution of sodium hydrosulphite before the soaping treatment in order to remove loosely attached dyestuffs from the surface of the textile material.

As examples of synthetic textile materials which can be coloured by the process of the invention there may be mentioned cellulose acetate and cellulose triacetate textile materials, polyamide textile materials, such as polycaprolactam and polyhexamethylene adipamide textile materials, polyalkylene textile materials such as polypropylene textile

materials, and preferably aromatic polyester materials such as polyethylene terephthalate textile materials. Such textile materials can be in the form of thread, yarn, webs, or woven or knitted fabric. If desired the synthetic textile materials can be in the form of unions with other textile materials, for example polyester/cotton or polyester/wool unions.

By the process of the invention synthetic textile materials are uniformly coloured in up to heavy depths of shade having excellent fastness to the tests commonly applied to such textile materials.

The invention is illustrated but not limited by the following Examples in which the parts and percentages are by weight:—

EXAMPLE 1

A woven polyester/cotton blended textile material (containing 67% of polyester and 33% of cotton) is padded through a liquor at 20°C comprising:—

	parts	
25% Aqueous dispersion of 3 - methyl - 4 - (6' - thiocyanato benzthiazol - 2 - ylazo) - N:N - di(β - acetoxyethyl) aniline	40	90
25% Aqueous solution of the sodium salt of partially hydrolysed polyacrylonitrile	20	95
Aqueous solution containing 14% 14% oleic acid and 29% of a sulphonated oleate	5	
Water	935	
	1000	100

and the textile material is then passed between rollers so that it only retains its own weight of the said liquor. The material is dried at 110°C. during 2 minutes and is then subjected to a dry heat treatment comprising baking for 1 minute at 180°C. at atmospheric pressure followed by 2 minutes at 180°C. under a pressure of 13 mms. of mercury.

The colored textile material is then rinsed in water, treated for 15 minutes in an aqueous solution at 50°C. containing 0.2% of sodium hydroxide and 0.2% of sodium hydrosulphite, rinsed in water, "soaped" for 10 minutes in a 0.2% aqueous solution of a synthetic detergent at 100°C., rinsed again in water and finally dried.

The textile material is colored a red shade having excellent fastness properties, and the polyester portion of the textile material is colored a deeper shade than the polyester portion of a similar textile material which was colored by the same method except that the dry heat treatment was carried out for

3 minutes at 180°C. at atmospheric pressure.

EXAMPLE 2

The procedure of Example 1 is repeated except that the 40 parts of the 25% aqueous dispersion of the dyestuff used in Example 1 are replaced by 40 parts of a 25% aqueous dispersion of 2 - nitrodiphenylamine 4 - sulphonanilide, and the baking treatment is carried out at a pressure of 52 mms. of mercury instead of at 13 mms. of mercury.

The textile material is thereby colored a bright yellow shade of excellent fastness properties, and the polyester portion of the textile material is colored a deeper shade than the polyester portion of a similar textile material which was colored by the same method except that the dry heat treatment was carried out for 3 minutes at 180°C. at atmospheric pressure.

EXAMPLE 3

A woven polyester/wool blended textile material (containing 50% of polyester and 50% of wool) is padded through a liquor at 20°C. comprising:—

	parts
25% Aqueous dispersion of 3 - methyl - 4 - (2 ¹ - cyano - 4 ¹ - nitrophenylazo) - N : N - di - (β - acetoxyethyl)aniline	40
25% Aqueous solution of the sodium salt of a partially hydrolysed polyacrylonitrile	20
Aqueous solution containing 14% oleic acid and 29% of a sulphonated oleate	5
Water	935
	1000

and the textile material is then passed between rollers so that it only retains its own weight of the said liquor. The material is dried at 110°C. during 2 minutes and is then subjected to a dry heat treatment comprising baking for 1 minute at 150°C. at atmospheric pressure followed by 2 minutes at 150°C. under a pressure of 13 mms. of mercury.

The colored textile material is then processed as described in the second paragraph of Example 1.

The textile material is colored a deep rubine shade having excellent fastness properties.

EXAMPLE 4

A woven cotton textile material is padded through a liquor at 20°C. comprising:

	parts
25% Aqueous dispersion of 1:4 - bis(3 ¹ - methyl - 4 ¹ - hydroxy - phenylazo) benzene	40
25% Aqueous solution of the sodium salt of a partially hydrolysed polyacrylonitrile	20
Aqueous solution containing 14% oleic acid and 29% of a sulphonated oleate	5
Water	935
	1000

and the textile material is then passed between rollers so that it only retains its own weight of the said liquor.

The textile material is dried at 110°C. and is then pressed in contact with a piece of undyed woven polyester textile material, and the whole then baked for 1 minute at 190°C. at atmospheric pressure followed by 2 minutes at 190°C. at a pressure of 13 mms. of mercury.

The polyester textile material is coloured a much deeper shade by this baking treatment than when the baking treatment is carried out for 3 minutes at 190°C. at atmospheric pressure.

A similar result is obtained when the cotton and polyester are used in the form of yarns which are twisted together before the baking treatment.

EXAMPLE 5

A glossy white art paper is printed, using an engraved roller, with a printing ink comprising:—

	parts
25% Aqueous dispersion of 2 - nitrodiphenylamine - 4 - sulphon - anilide	40
10% Aqueous solution of a starch-ether thickening agent	35
Water	25
	100

and the paper is then dried. The paper is then placed in contact with a woven filament polyester fabric, which is then heated for 10 seconds at 190°C. under a pressure of 70 mms. of mercury. The polyester fabric is thereby printed in a yellow shade, and the colour transfer obtained is much higher than that obtained when the heating is carried out for 15 seconds at 210°C at atmospheric pressure.

WHAT WE CLAIM IS:—

1. Process for colouring synthetic textile materials or unions containing synthetic tex-

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3 minutes at 180°C. at atmospheric pressure.

EXAMPLE 2

The procedure of Example 1 is repeated except that the 40 parts of the 25% aqueous dispersion of the dyestuff used in Example 1 are replaced by 40 parts of a 25% aqueous dispersion of 2 - nitrodiphenylamine 4 - sulphonanilide, and the baking treatment is carried out at a pressure of 52 mms. of mercury instead of at 13 mms. of mercury.

The textile material is thereby colored a bright yellow shade of excellent fastness properties, and the polyester portion of the textile material is colored a deeper shade than the polyester portion of a similar textile material which was colored by the same method except that the dry heat treatment was carried out for 3 minutes at 180°C. at atmospheric pressure.

EXAMPLE 3

A woven polyester/wool blended textile material (containing 50% of polyester and 50% of wool) is padded through a liquor at 20°C. comprising:—

	parts
25% Aqueous dispersion of 3 - methyl - 4 - (2 ¹ - cyano - 4 ¹ - nitrophenylazo) - N:N - di - (β - acetoxyethyl)aniline	40
25% Aqueous solution of the sodium salt of a partially hydrolysed polyacrylonitrile	20
Aqueous solution containing 14% oleic acid and 29% of a sulphonated oleate	5
Water	935
	1000

and the textile material is then passed between rollers so that it only retains its own weight of the said liquor. The material is dried at 110°C. during 2 minutes and is then subjected to a dry heat treatment comprising baking for 1 minute at 150°C. at atmospheric pressure followed by 2 minutes at 150°C. under a pressure of 13 mms. of mercury.

The colored textile material is then processed as described in the second paragraph of Example 1.

The textile material is colored a deep rubine shade having excellent fastness properties.

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25% Aqueous solution of the sodium salt of a partially hydrolysed polyacrylonitrile	20
Aqueous solution containing 14% oleic acid and 29% of a sulphonated oleate	5
Water	935
	1000

and the textile material is then passed between rollers so that it only retains its own weight of the said liquor.

The textile material is dried at 110°C. and is then pressed in contact with a piece of undyed woven polyester textile material, and the whole then baked for 1 minute at 190°C. at atmospheric pressure followed by 2 minutes at 190°C. at a pressure of 13 mms. of mercury.

The polyester textile material is coloured a much deeper shade by this baking treatment than when the baking treatment is carried out for 3 minutes at 190°C. at atmospheric pressure.

A similar result is obtained when the cotton and polyester are used in the form of yarns which are twisted together before the baking treatment.

EXAMPLE 5

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Water	25
	100

and the paper is then dried. The paper is then placed in contact with a woven filament polyester fabric, which is then heated for 10 seconds at 190°C. under a pressure of 70 mms. of mercury. The polyester fabric is thereby printed in a yellow shade, and the colour transfer obtained is much higher than that obtained when the heating is carried out for 15 seconds at 210°C at atmospheric pressure.

WHAT WE CLAIM IS:—

1. Process for colouring synthetic textile materials or unions containing synthetic tex-

tile materials which comprises applying a water-insoluble dyestuff free from fibre-reactive groups to the textile material and the
5 subjecting the textile material to a dry heat treatment at a pressure less than atmospheric pressure.

2. Process as claimed in Claim 1 wherein the dry heat treatment is carried out at a pressure of between 0.1 and 100 mms. of
10 mercury.

3. Process as claimed in Claims 1 or 2 wherein the dry heat treatment is carried out at a temperature between 110° and 240°C.

4. Process as claimed in any of Claims 15 1 to 3 wherein a part of the dry heat treatment is carried out at a pressure less than atmospheric pressure and the remainder is carried out at atmospheric pressure.

5. Process as claimed in any of Claims 20 1 to 4 wherein there is additionally used a dyestuff for any non-synthetic textile material present.

6. Process as claimed in any of Claims 25 1 to 5 wherein the dyestuff is applied to the textile material by a dyeing, padding or printing method.

7. Modification of the process as claimed in Claim 1 wherein a print paste containing the water-insoluble dyestuff is printed
30 onto a cellulose material, which is optionally dried, and is then placed in contact with the synthetic textile material and the whole subjected to a dry heat treatment at a pressure less than atmospheric pressure.

35 8. Process as claimed in Claim 7 wherein

the print paste is a water-based or oil-based ink.

9. Process as claimed in Claims 7 or 8 wherein the cellulose material is paper.

10. Process as claimed in any of Claims 40 7 to 9 wherein the dry heat treatment is carried out at a pressure of between 0.1 and 100 mms of mercury.

11. Process as claimed in any of Claims 45 7 to 10 wherein the dry heat treatment is carried out at a temperature between 110° and 240°C.

12. Process as claimed in any of Claims 50 7 to 11 wherein the printed cellulose material and the synthetic textile material are passed together between heated rollers, the pressure round the rollers being maintained at less than atmospheric pressure.

13. Process as claimed in any of Claims 55 1 to 12 wherein the synthetic textile material is an aromatic polyester textile material.

14. Process as claimed in Claim 13 wherein the polyester textile material is a polyethylene terephthalate textile material.

15. Process as claimed in any of Claims 60 1 to 14 as hereinbefore particularly described especially with reference to any of the Examples.

16. Synthetic textile materials whenever coloured by a process as claimed in any of
65 Claims 1 to 15.

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PATENTS ACT 1949

SPECIFICATION NO 1271372

In accordance with the Decision of the Principal Examiner, acting for the Comptroller-General, dated 17 December 1974 this Specification has been amended under Section 33 in the following manner:-

Page 3, after line 109 insert Our attention has been drawn to the publication of Specification No 830348 disclosing a different kind of dyeing process carried out in high vacuum. We do not claim any process carried out in high vacuum or at a pressure of less than 0.1 mm of mercury. Subject to this disclaimer

Page 4, delete 27, 28 and 29 insert Process for colouring synthetic textile materials wherein a print paste containing a water-insoluble dyestuff free from fibre-reactive groups is printed

THE PATENT OFFICE
5 February 1975

R 20262/22

