

UNITED STATES PATENT OFFICE

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DYEING OR PRINTING

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This invention relates to dyeing and printing and more particularly to the development of the colour from soluble ester derivatives of reduced vat dyestuffs which eliminate the use of vats, such as are to be found for instance described in British Patents Nos 186,057, 202,630, 202,632, 203,681, 218,649, 231,159, 231,889, 234,829, 245,587, 247,787, 248,802, 251,491, 258,626, 260,638, 260,647, 261,139, 263,898, some of these specifications indicating the use of an acid bath for colour development.

Heretofore, the methods mostly employed for the development of the colour on the fibre consist in treating the dyed or printed or padded fabric with or without a catalyst in the presence of an acid oxidizing agent for example in a dye-bath with a nitrate and an acid, ferric chloride and an acid or bichromate and an acid. In the case of printed or padded goods the development was effected usually by incorporating in the padding liquor an oxidising agent, such as chlorate with or without a catalyst, such as potassium ferricyanide and developing the colour by steaming.

In the case of certain dyestuffs the development of colour is to some extent difficult to control on the above lines, owing apparently to a tendency for the dyestuff to become over oxidized. This is particularly the case with indanthrones. Hitherto, indanthrones the most important examples of the anthraquinone vat colours have not been able to be developed by the oxidizing agents already disclosed because of the fact that these oxidizing agents cause over-oxidation.

The object of the present invention is to provide a process free of these disadvantages or offering a useful alternative, that is to say an improved or modified process for the development of the colour after the soluble derivative has been deposited on or absorbed by the fibre as distinct from the application of the soluble derivative to the fibre.

To that end, we have made experiments and we have found that colour development can be obtained by treating the dyed fabric preferably at high temperatures for example at the boil with a dilute acid with the addition of a small amount of a cupric salt, for example cupric sulphate or cupric chloride.

The process is specifically advantageous when used with the indanthrones. These dyes could not be developed successfully with the aid of any of the oxidizing agents already used, and the use of the copper salt has enabled them to be applied in this manner for the first time.

The invention in brief consists in a process for the development of indanthrone colours by means of dilute aqueous acid solutions, in materials dyed, printed or the like with ester derivatives of reduced indanthrone, which eliminate the use of vats, such as are described, for instance, in the specifications referred to by number above, in which the development is carried out in the presence of a cupric salt, preferably cupric sulphate, at elevated temperatures, for example, at boiling temperatures, preferably in the presence of acid, for instance, sulphuric acid.

The invention also consists in methods for the development of colours herein described and any products which may be produced thereby.

The following examples illustrate how the invention may be carried into effect, all parts being parts by weight:—

Example 1

Wool is dyed with the water soluble ester of dimethoxy-dibenzanthrone prepared according to British Patent No. 247,787. It is then rinsed and immersed in a bath at 100° C. containing 5 parts of cupric sulphate and 10 parts of sulphuric acid per 1000 parts of water. The development bath is maintained at the boil the wool being worked in the usual

way. After about 10 minutes, the development is complete, the wool is withdrawn from the bath, well rinsed and treated with a dilute solution of neutral soap at 60° C., washed and dried. The development of the dyestuff may be carried out in the original dye liquor by adding sufficient sulphuric acid to make the solution up to 1 per cent. together with cupric sulphate. 10 parts per 1000 parts of hydrochloric acid or 10 parts per 1000 parts of formic acid can be used instead of the 10 parts per 1000 parts of sulphuric acid.

Example 2

In the above example, silk may be used instead of wool.

Example 3

Wool is dyed with the water soluble ester of indanthrone prepared according to British Patent No. 247,787. It is then developed at the boil on the lines of Example 1 in a bath containing 1 per cent. sulphuric acid and 0.5 per cent. of copper sulphate for about 10 minutes, until the correct shade is obtained. This method appears readily to avoid over oxidation and formation of yellow oxidation products of indanthrone.

Example 4

In the above Example 3, silk may be used instead of wool.

Example 5

Wool or silk is printed with a paste made up from 80 parts of British gum thickening and 20 parts of Soledon jade green (sulphuric acid esters of Caledon jade green, colour index No. 1101), dried, steamed and developed in a solution containing 10 parts of Glauber's salt, 10 parts of sulphuric acid and 5 parts of copper sulphate per 1000 parts, at the boil for about 5 minutes.

Example 6

Cotton is padded in a liquor containing 10 parts of Soledon jade green, 10 parts British gum thickening and 1 part soluble oil per 100 parts. The padded cloth is dried, steamed and developed in a solution containing 5 parts cupric sulphate and 10 parts of acetic acid per litre at the boil, for about 10-15 minutes, till the colour is developed.

Example 7

Cotton yarn is dyed with the water soluble ester, prepared according to British Patent No. 251,491, of the indanthrone derivative known as Caledon blue 3G (Supplement to the colour index dated January, 1928, published by the Society of Dyers and Colourists, page 16, item No. 1109). It is then developed by treating for a few seconds in a boiling bath of 1 per cent. sulphuric acid containing ½ per cent. of copper sulphate and 10 per cent. of common salt.

Example 8

According to this example, a chemically equivalent amount of cupric chloride is employed instead of cupric sulphate, in conjunction with 1 per cent. sulphuric acid or 1 per cent. hydrochloric acid or other suitable acid.

General

In the case of boiling with acid alone, which as is known also causes development of the dyestuff derivative, the period of boiling is comparatively long and as the strength of the acid is usually high, the fibre itself may be attacked. We find however that the addition of cupric salts enables good development of colour to be obtained in a much shorter period.

Examples of suitable acids are dilute sulphuric acid and dilute hydrochloric, formic and acetic acids, but the selection of the acid should be made having regard to the particular fibre it is desired to colour.

Colour development may be caused to proceed slowly or rapidly according to the amount and nature of the cupric salt, the concentration of the acid and the time of heating, so that the capacity of controlling the reaction is available. This is of advantage in the case of dyestuffs which are liable to over oxidation when in the presence of ordinary oxidizing agents, because the treatment may be stopped after the time necessary to convert the ester derivative into the dyestuff and before the dyestuff itself begins to oxidize.

The method is not limited to use with any particular fibres, but it is particularly suitable for use with animal fibres which suffer little or no harm from treatment in hot acid baths. With vegetable fibres, the necessary precautions must be taken to avoid the harmful effect of acid on the fibre.

The method may be used for the development of soluble ester derivatives of reduced vat dyestuffs in connection with dyed, printed or padded fabrics, or textile fibres.

Where in this specification and claims reference is made to dyed materials, this includes materials dyed, printed or padded.

Having now described our invention, what we claim as new and desire to secure by Letters Patent is:—

1. The process which consists in applying an enolic ethereal salt of indanthrone to a material, and then developing the colour by means of a cupric salt.

2. A process as claimed in claim 1 in which the cupric salt is a cupric salt of the group consisting of cupric sulphate and cupric chloride.

3. A process as claimed in claim 1, in which the development treatment is also carried out in the presence of an acid.

4. A process as claimed in claim 1, in which

the cupric salt is a cupric salt of the group consisting of cupric sulphate and cupric chloride, and the development process is carried out in the presence of an acid.

5 5. A process as claimed in claim 1, in which the development process is carried out in the presence of sulphuric acid.

6. Materials coloured by the process of claim 1.

10 7. A process as claimed in claim 1, carried out at substantially boiling temperature.

In testimony whereof we have signed our names to this specification.

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