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TREATMENT OF MATERIALS

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The present invention relates to the treatment of materials, which consist of or contain cellulose esters or ethers, in order to increase the fastness of colourations thereon.

Cellulose acetate artificial silk which has been coloured by dyeing or printing, is known to be liable to become affected when exposed to an impure atmosphere such as that produced when coal gas is burned in an imperfectly ventilated space, in such a way that the coloured parts of the material fade. This trouble is encountered, for example, when the material is stored in a warehouse where coal gas is burned. It will be understood that particularly those parts of the material suffer injury, which come into direct contact with the atmosphere carrying the fumes resulting from the combustion of the coal gas. Thus in the case of piece goods the edges will fade or indicate change of shade. In this manner the material as a whole is disfigured and its value diminished; indeed the material may be be spoiled as to become unsaleable.

Whilst cellulose esters and ethers which have been dyed, are generally subject to the aforesaid fading, the latter is particularly liable to occur when the dyestuffs which have been employed are derivatives of anthraquinone, especially when the shades in question are blue or violet.

We have now found that the above-described fading can be considerably retarded, or indeed prevented, by the application to the material of organic compounds which are amidines as hereinbelow defined.

According to the invention material consisting of or containing esters or ethers of cellulose is treated with a derivative of formamidine, in which each nitrogen atom bears a phenyl substituent which itself may carry simple groups or atoms such as alkyl, alkoxy or halogen, and in which the two remaining hydrogen atoms of the formamidine molecule may be replaced each or both by an alkyl group containing not more than four carbon atoms.

Accordingly there may be employed in accordance with the present invention, for example, NN'-diphenylformamidine

$$(C_6H_5—NH—CH=N—C_6H_5)$$

NN'-diphenylacetamidine

$$(C_6H_5—NH—C(CH_3)=N—C_6H_5)$$

N-methyl-NN'-diphenylacetamidine

$$(C_6H_5—N(CH_3)—C(CH_3)=N—C_6H_5)$$

N-ethyl-NN'-diphenylacetamidine

$$(C_6H_5—N(C_2H_5)—C(CH_3)=N—C_6H_5)$$

and N-phenyl-N'-p-tolyl-N-methylacetamidine

$$(C_7H_7—N(CH_3)-C(CH_3)=N—C_6H_5)$$

N-methyl-N:N'-diphenylacetamidine is made by treating with N-methylaniline the product of reacting acetanilide in benzene with phosphorus pentachloride. N-phenyl-N'-p-tolyl-N-methylacetamidine is obtainable by condensing acet-p-luidine with N-methylaniline in phosphorus trichloride.

These amidines may be applied to the dyed material consisting of or containing cellulose esters or ethers in any convenient medium. The amidines may be used in the form of solutions or dispersions. The application of the amidine is conveniently effected by dipping the material it is desired to treat, into an aqueous dispersion of the desired amidine, squeezing out most of the adhering liquid and drying. The amidines may likewise be applied in solution in alcohol. The amidines must, however, be used in the form of the free bases. The use of salts of amidines is not included in the invention. The material may also be treated with the amidine in the manner just described during the dyeing thereof, but the amidine is preferably applied after the process of dyeing, that is, to the already dyed material.

Under ordinary storage conditions the fading occasioned by coal gas fumes develops slowly and more or less prolonged storage, depending on the state of the atmosphere and the amount of obnoxious gas present, is necessary before noticeable fading occurs, but fading for testing purposes may be accelerated for instance by exposure to the fumes of burning coal gas in a restricted space.

For this purpose the dye patterns which it is required to submit to the action of the fumes, may be suspended within the upper end of an iron pipe, conveniently 5–8 feet high and six inches in diameter, which is disposed vertically above a lighted gas burner in such a manner that the hot air arising from the burner and laden with the fumes of the burnt gas, is guided upwards and caused to impinge upon the patterns. Any form of apparatus embodying a like principle and designed so that the material to be tested can be immersed in an atmosphere comprising the products of combustion of the burning gas, may likewise be employed. In such an apparatus it is found that dyed material which is liable to fade in contact with the fumes of burning gas, is noticeably impaired in shade in the course of several hours, for instance, 6–10 hours.

If patterns of dye cellulose acetate artificial silk fabric which have been treated with amidines according to the present invention, are exposed in such an apparatus as described above, side by side with the patterns of similarly dyed, untreated material, it is apparent that the treated material is possessed of considerably enhanced re-

sistance to the fading action of the gas fumes, compared with the untreated material.

As has already been indicated above, the material which may be treated according to the invention is not confined to fabrics consisting wholly of cellulose esters or ethers, for example, cellulose acetate artificial silk fabric, but includes also, for instance, mixed fabrics of viscose and cellulose acetate artificial silks.

The following examples, in which the parts are by weight, illustrate but do not limit the invention.

Example 1

10 parts of NN'-diphenylacetamidine are pebble-milled with 90 parts of water and ½ part of a 50% aqueous solution of the sodium salt of the condensation product from β-naphthalenesulphonic acid and formaldehyde. At the end of 24 hours, a fine dispersion has been produced. The dispersion is then diluted with water to a strength of 1%.

A piece of cellulose acetate artificial silk fabric which has been dyed with 1% by weight of 1:4:5:8-tetraamino-anthraquinone is divided into two portions. One portion is immersed for one minute at 15–20° C. in the above described aqueous dispersion of NN'-diphenylacetamidine. The fabric is then squeezed so that it retains approximately its own weight of liquid, and dried.

The portion of fabric which has been treated with NN'-diphenylacetamidine is exposed together with the untreated portion for 10 hours to the fumes of burning coal gas in an apparatus such as that previously described. At the conclusion of the test the untreated portion of the fabric is found to have considerably faded, the shade having become much duller, whereas the shade of the treated fabric has been affected only to a slight degree.

Instead of employing an aqueous dispersion of NN'-diphenylacetamidine, the latter may be applied to the fabric in the form of a 1% (weight by volume) solution in ethyl alcohol, with similar results.

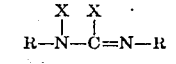
Example 2

A pattern of cellulose acetate artificial silk which has been dyed with 1% by weight of 1:4:5:8-tetraaminoanthraquinone is immersed at about 20° C. in a 1% aqueous dispersion of N-methyl-NN'-diphenylacetamidine, which has been prepared in the same manner as the amidine dispersion described in Example 1. After the fabric has been immersed for one minute it is removed from the dispersion, squeezed until it holds about its own weight of liquid and then dried. When the so treated fabric is exposed to the fumes from burning coal gas, by the method described in the previous example, it suffers only a slight change in shade; on the other hand, when the untreated fabric is exposed to gas fumes under the same conditions, bad discolouration results.

In place of N-methyl-NN'-diphenylacetamidine, there may be employed with similar effect N-phenyl-N'-p-tolyl-N-methylacetamidine.

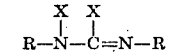
We claim:

1. The process for preventing or retarding the fading of dyes on cellulose acetate when such colored materials are subjected to gas fumes, which comprises treating said cellulose acetate material with an N-N'-diarylformamidine compound of the formula:



in which each R stands for an aryl radical of the group consisting of phenyl and tolyl radicals, and where each X stands for a substituent of the group consisting of —H and alkyl groups containing not more than 4 carbon atoms.

2. Cellulose acetate material which is dyed with a color that is not fast to gas fumes on such material but which color on said material has been rendered fast to such fumes by having incorporated in the material an N-N'-diarylformamidine compound of the formula:



in which each R stands for an aryl radical of the group consisting of phenyl and tolyl radicals, and where each X stands for a substituent of the group consisting of —H and alkyl groups containing not more than 4 carbon atoms.

3. Cellulose acetate material which is dyed with a color that is not fast to gas fumes on such material but which color on said material has been rendered fast to such fumes by having incorporated in the material N-N'-diphenylformamidine.

4. Cellulose acetate material which is dyed with a color that is not fast to gas fumes on such material but which color on said material has been rendered fast to such fumes by having incorporated in the material N-N'-diphenylacetamidine.

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