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COLORATION OF TEXTILE MATERIALS

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This invention relates to improvements in coloring operations and is more particularly concerned with the coloration of textile and similar materials by the oxidation on the material of aromatic amines, i. e., with the aniline black type of coloration.

As is well known, black and various other colors, including greys, browns, fawns and yellows, have for a long time been produced on textile and other materials by the oxidation of aromatic amines, including aniline, paraphenylene diamine, para-amino-diphenylamine, para-amino-phenol and the like. Such colorations are referred to as "aniline black type of colorations" in the present specification. The material is commonly impregnated with an aqueous liquid containing the aromatic amino compound together with an oxidising agent which becomes active under heat, for example a chlorate, and usually a catalyst, such as compounds of copper, manganese, iron, vanadium or cerium. The impregnation may be effected by bath treatment methods or by printing or padding. The oxidation may be effected in the same bath, where a bath treatment is used, or in a subsequent ageing or steaming treatment. When using the ageing process an afterchroming is commonly applied, for example a simple treatment with a bichromate solution with or without a small proportion of sulphuric or other acid. In the ageing process the goods which have been impregnated with the aqueous liquor containing the aromatic amine, the oxidising agent and the catalyst, are customarily dried and then subjected to the ageing or steaming treatment. During the drying operation there is a tendency for the ingredients of the liquor to migrate during the evaporation of the water, with the result that the goods may be impregnated with the ingredients in a non-uniform manner at the end of the drying operation so that the resultant coloration is itself non-uniform. It is a common practice to include in the liquor gum arabic or other thickening agent to minimise this tendency. When, however, it is attempted to carry out this type of coloration on goods which are in wound package form, as for example yarns on bobbins or fabrics wound on beams, the addition of a thickening agent is a serious disadvantage in that it tends to prevent rapid penetration of the goods by the liquor and, moreover, it appears to interfere somewhat with the drying operation.

We have now found that in this type of coloration very rapid penetration may be achieved and at the same time the impregnation is very uniform if the liquid medium of the impregnation liquor applied to the goods contains a substantial proportion of a volatile organic liquid, for example acetone, the lower boiling alcohols and ethers, dioxane, methylene ethylene ether and the like. Not only is the penetration of a wound package much quicker than is the case when using a purely aqueous liquor, but in addition the drying operation itself is very much shortened, and, as already indicated, there is little or no tendency for the ingredients of the liquor to migrate during the drying operation.

According to the present invention therefore, in the production of an aniline-black type of coloration on a textile material, the material is impregnated with a liquor containing the requisite aromatic amine and a substantial proportion of a volatile organic liquid, and the aromatic amine thereafter oxidised to the coloring matter on the material. Preferably the oxidation color ingredients are applied together from a common medium. Thus, the material may be impregnated with a liquor containing aniline, a chlorate, an oxidation catalyst, and a substantial proportion of a volatile organic liquid, and then aged. In some cases an increase in solubility of the ingredients in the liquid medium of the impregnation liquor may be brought about by incorporation of water therein. However, the water is preferably not used in a proportion which would seriously retard the drying. Thus, for example, the amount of water may be and preferably is so chosen as to be smaller than that amount which would evaporate normally in the same time as the organic liquid present under the drying conditions used. For example, in the case of using acetone, an amount of water may be present equal to about one quarter of the weight of acetone when the drying operation is carried out at 10–20° C. Similar considerations apply to other organic liquids. In some cases the organic liquid may form an azeotropic mixture with the water, which again facilitates evaporation. In this case also the water is preferably present in a proportion equal to or smaller than that corresponding with the azeotropic mixture under the drying conditions.

The liquid medium of the impregnation liquor may contain 40, 60, 80% or more of volatile organic liquid according to circumstances.

In some cases where difficulty is found in selecting a single organic liquid to dissolve all the ingredients which it is desired to apply, a mixture of two or more organic liquids with or without water, as previously explained, may be used,

or alternatively a plurality of organic liquids may be used successively, some of the ingredients being dissolved in one liquid and some in others. However, application of all the ingredients in a single operation is to be preferred.

The ingredients for the production of the oxidation color may be those commonly employed in the art. For example the aromatic amines may be those specified above. As the oxidising agent a chlorate, and particularly sodium or potassium chlorate, is preferred. The oxidation catalyst may, for example, be copper sulphate, cupric chloride, potassium ferrocyanide, ammonium vanadate or the like. In the production of oxidation colors on cellulosic materials, for example cotton or other natural cellulosic fibres, viscose or other regenerated cellulose artificial silks and the like, the aniline or other aromatic amine may be applied in the form of a mineral acid salt, for example, the hydrochloride, but it is sometimes better to use a mixture of mineral acid salt of the base together with free base, or in other words to use hydrochloric acid or other mineral acid in admixture with aniline or other aromatic base in a proportion less than is chemically equivalent to the base. In the case of producing oxidation colors on cellulose acetate or other cellulose derivatives, it is, on the other hand, preferable to employ a mineral acid in a proportion equivalent to the aniline or other aromatic base, and, in addition, to have present a mineral or preferably an organic acid or an acid reacting substance in excess. This process is described in detail in U. S. Patents Nos. 1,927,160 and 1,927,161. As therein indicated, glacial acetic acid and formic acid are very suitable examples of an acid substance to have present in addition to the mineral acid equivalent to the aromatic base.

The new process is applicable to the dyeing of various textile materials. It may be applied to regenerated cellulose artificial silk, for example, that prepared from viscose or by saponifying cellulose-ester artificial silk, including cellulose-ester artificial silk which has been stretched in steam or hot water or in presence of organic softening agents. Again cellulose-ester or -ether materials may be colored, for example, materials of cellulose acetate, cellulose formate, butyrate or propionate or materials of methyl, ethyl, butyl, propyl or benzyl cellulose. It will be understood that in any particular case the liquid medium of the impregnation liquor will be selected with due regard to the solubility properties of the material to be treated and the substances to be applied thereto.

The following example illustrates the invention:

Regenerated cellulose yarn is wound on a bobbin with a perforated barrel and impregnated with the following liquor.

	Per cent
Aniline hydrochloride	10
Sodium chlorate	3.5
Cupric chloride	0.3
Water	17
Acetone	69

The impregnation is effected by circulating the liquid through the windings on the bobbin either under pressure or under suction, and preferably the direction of flow is reversed occasionally. The circulation is continued for 15 minutes in the cold. After the impregnation the bobbin is centrifuged to remove excess liquor and is dried by connecting the interior to a vacuum for half an hour. Oxidation is then effected by sucking air through the bobbin in a damp atmosphere for about half an hour at 40° C. Finally the bobbin is treated for half an hour in the cold in a 0.5% solution of sodium bichromate and is then washed, treated with a 0.2% solution of sodium carbonate and washed again. The yarn is dyed a good black shade fast to washing.

In a similar way cellulose acetate yarn may be dyed black on the bobbin. The organic liquid should be so chosen that the liquid medium of the impregnation liquor dissolves the material to be applied to the yarn but does not dissolve the yarn and preferably so that it has no pronounced swelling action upon the yarn. Thus, the oxidation color ingredients may be applied to cellulose acetate materials from lower aliphatic alcohols or their aqueous mixtures. For example, a mixture of 3 parts methylated spirits with 1 part of water may be used.

While the invention has been described above more particularly with reference to the dyeing of material in wound package form and it is in this connection that the advantages are most apparent, the invention is not limited thereto but includes the dyeing of material in any desired form. Thus the invention is applicable to the coloration of running threads and other processes in which the materials are passed through the dye liquor for a short time only. In such processes, media having a swelling action on the materials treated are preferably employed in order to obtain rapid impregnation.

Having described our invention what we desire to secure by Letters Patent is:

1. Process for the production of an aniline b'ack type of coloration on textile material, which comprises impregnating the material in wound package form with a liquor containing the requisite aromatic amine at least partially in the form of a salt, water, an oxidizing agent which becomes active when heated and about 60 to 80% of a water-miscible volatile organic liquid, and thereafter drying and ageing the impregnated material, the proportion of water in the impregnation liquor being such that it is wholly evaporated off before the organic liquid constituent.

2. Process for the production of aniline black on a cellulose textile material, which comprises impregnating the material in wound package form with a liquor containing aniline at least partly as a salt, water, a copper salt, and a chlorate, the liquid medium of the liquor being a mixture of about 60 to 80% of acetone with 40 to 20% of water, and thereafter drying and ageing the impregnated material.

3. Process for the production of aniline black on a cellulose acetate textile material, which comprises impregnating the material in wound package form with a liquor containing aniline at least partly as a salt, water, a copper salt, and a chlorate, the liquid medium of the liquor being a mixture of about 60 to 80% of alcohol with 40 to 20% of water, and thereafter drying and ageing the impregnated material.

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