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PROCESS OF DYEING CELLULOSE ACETATES.

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To all whom it may concern:

Be it known that I, RENÉ CLAVEL, a citizen of the Swiss Republic, of Basel, Switzerland, have invented a certain new and useful Improved Process of Dyeing Cellulose Acetates, of which the following is a specification.

This invention relates to the dyeing of cellulose acetates in artificial silk, film or other form with development dyes or so-called ice colours.

It has been proposed to produce azo dyes on cellulose acetate goods by development on the fibre or material (see for example U. S. Patent 1,002,408 and British Patent 24284 of 1907).

In the technical application of the development dyes or so-called ice colours for dyeing cellulose acetates, such for example as cellulose acetate silks, fabrics, films or other goods, it has been found that practical dyeing to sample or standard is connected with some difficulties, inasmuch as after the application of the base to the cellulose acetate silk or goods it has been necessary to take out a specimen hank or skein or other corresponding specimen for diazotizing and developing. The dyeings so obtained were then compared with the sample, and according as they turned out, the base or the developing bath had to be strengthened or weakened. This sampling or matching required time, was rather difficult to perform, and moreover required skilled dyers.

In the present invention the cellulose acetate goods are dyed with development dyes by any of the following combinations of baths: (*a*) employing the bases and developers in one bath and diazotizing in a second bath, or (*b*) applying the base in one bath, diazotizing in the same bath and developing in a second bath, or (*c*) applying the base or developer in the first bath and diazotizing and coupling in the second bath, and according to the invention, in effecting the dyeing of the goods by any of these combinations of baths, I employ protective colloids, such as boiled-off liquor (i. e. the liquor resulting from the boiling-off or degumming of raw natural silk by boiling with soap solutions gelatine, and so forth to avoid precipitation or separation of bases, developers or dyestuffs by salting out.

In any case it is seen that application of two of the dye-forming factors: base, diazotization, and developer occurs in one bath and that of the third factor in another bath. These various baths may be employed with or without addition of a diazotizable or non-diazotizable dyestuff, and with or without acids and salts or any of these, but a protective colloid will always be employed in at least one of the baths.

It has been found that it is possible to work very quickly and well if one applies the base and developer in one bath, and if necessary adds a dyestuff according to the shade required, and then diazotizes in a second bath, or if one applies the base, if necessary with a dyestuff, in one bath and after some time adds to this bath the calculated or required quantity of nitrite and acid and then couples with a developer in a second bath; the added dyestuff may be diazotizable or non-diazotizable. Or one may work by impregnating the acetyl-celluloses either with the base or with the developer, and if desired, with a dyestuff, and afterwards diazotizing and coupling in a second bath.

Further, there may be added to the baths, in order to facilitate rapid absorption or taking up by the goods, acetic, lactic or other organic or inorganic acids or water-soluble salts, preferably chlorides, or both acids and salts.

In the first case, (*a*), where we have base and developer in one bath, the operation may be as follows:—

The cellulose acetates, for example in the form of hanks or skeins, are introduced into the bath containing the base, developer and protective colloid and if desired an added dyestuff and turned therein. After sufficient dyeing up, a small specimen hank or skein is removed from the bath, washed quickly and diazotized, the diazotizing bath consisting for example of sodium nitrite and hydrochloric acid. If the dyed shade corresponds to the sample hank or skein the batch is washed and diazotized; if it does not, more base or more developer or dyestuff is added to the before mentioned bath according to requirement, until a specimen skein corresponds to the requirements.

In the second case (*b*), one may operate by first treating the goods with the base alone or together with a dyestuff, in the base bath. After moving the goods in the bath for some time, a specimen hank is withdrawn, diazotized and developed, and according to requirement one then adds more base or more dyestuff to the bath, afterwards diazotizing in the bath and developing in a

second bath as before mentioned, a protective colloid being used in either or both of the baths.

It has been found that no dimming or dulling of the acetate silk dyeing occurred by the addition of a basic diazotization-resisting dyestuff to the base- or developer-bath, or base and developer bath, whereas according to previous processes some dimming or dulling of the fibre has sometimes been caused by the diazotizing.

As before mentioned one may also operate according to the third case, (*c*), by impregnating the cellulose acetate first in the developer- or base-bath, washing, and then transferring to the base-diazotization or developer-diazotization bath, a protective colloid being used in either or both baths.

Example 1.

In order to dye 1 kg. of cellulose acetate silk a brown-red, the goods are introduced into a bath consisting of 25 liters water, 20 gr. amidoazobenzene, 30 gr. betanaphthol, 5 gr. Capri blue, 2 liters boiled-off liquor. The goods are treated for half an hour at ordinary temperature, and then 50 grams magnesium chloride are added and the bath is heated to about 60° C. After a further half hour, during which the cellulose acetate silk is repeatedly turned in the bath, the goods are washed and transferred to the diazotizing bath. This consists of 20 liters water, 50 gr. sodium nitrite, 100 cc. concentrated hydrochloric acid. The goods are kept moving in the bath for half an hour at a low temperature. Then follow one washing with hard water and one with soft water, and then a soap bath at 60° C., followed by one washing with soft water. This is followed by the usual brightening bath.

Example 2.

1 kg. of cellulose acetate silk is introduced into a bath consisting of 25 liters water, 20 gr. amidoazotoluene, 2 liters boiled-off liquor. The bath is heated at 60° C. and the goods are turned in it for about half an hour. Then 50 cc. technical sulphuric acid and 50 grams sodium nitrite are added to this bath and the goods are diazotized with repeated turning in the bath. After a further half hour the acetate silk is washed and entered into the developing bath at 60° C consisting of 25 liters water, 30 gr. betanaphthol, 50 gr. soap. After an hour the goods are washed and brightened.

What I claim and desire to secure by Letters Patent is:—

1. In a process for dyeing cellulose acetates in artificial silk, film or other form by production of development dyes on the material, applying two of the three dye-forming factors: base, diazotizing means, and developer in one bath and the other factor in another bath, a protective colloid being employed in at least one of said baths.

2. In a process for dyeing cellulose acetates in artificial silk, film or other form by production of development dyes on the material, applying two of the three dye forming factors: base, diazotizing means, and developer in one bath and the other factor in another bath, a protective colloid being employed in at least one of said baths, and an added dyestuff being employed in at least one of the baths.

3. In a process for dyeing cellulose acetates in artificial silk, film or other form by production of development dyes on the material, applying two of the three dye-forming factors: base, diazotizing means, and developer in one bath and the other factor in another bath, a protective colloid being employed in at least one of said baths, and an acid being also employed in at least one of said baths.

4. In a process for dyeing cellulose acetates in artificial silk, film or other form by production of development dyes on the material, applying the base and developer in one bath containing protective colloid, and diazotizing in a second bath.

5. In a process of dyeing cellulose acetates in artificial silk, film or other form by the production of development dyes on the material applying the base and developer in one bath containing protective colloid, with addition of a dyestuff, and diazotizing in a second bath.

6. In a process of dyeing cellulose acetate in artificial silk, film or other form by the production of development dyes on the material, applying the base and developer in one bath containing protective colloid, with addition of an acid, and diazotizing in a second bath.

In testimony whereof I have hereunto subscribed my name.

DR. RENE CLAVEL.