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PROCESS OF DYEING AND PRINTING CELLULOSE ESTERS

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The problem of producing dyeings with vat dyestuffs on cellulose acetate (cellulose ester) has not yet been solved. There have been, of course, made attempts to overcome the existing difficulties, see for instance the German specification No. 415,937. All these attempts, however, did not give—in view of the requirements of the practice—satisfactory results. Also with the aid of ester salts of vat dyestuffs, such as they are for instance obtained according to the process described in the U. S. specification No. 1,448,251, when applied on cellulose acetate according to the process of the U. S. specification No. 1,575,958, it has not been possible up to-date to obtain the desired results. The cellulose acetate will not be sufficiently penetrated by the solutions of the ester salts; when developed by oxidation the dyestuff is merely precipitated on the surface of the fibre; the dyeings are not solid to washing and rubbing, i. e. they are worthless.

Now I have found a suitable way for obtaining useful dyeings and printings on cellulose acetate with these ester salts by modifying the process disclosed in the U. S. specification No. 1,575,958. According to my invention cellulose acetate is padded with a solution of an ester salt of a vat dyestuff, then wrung, dried and steamed. In this way the ester salt is fixed on the fibre as such, so that the fibre can be rinsed or even soaped without substantial loss of ester salt. Thereupon the dyeing is developed in the usual manner by way of oxidation (by means of sulphuric acid and nitrite, or suphuric acid and bichromate). In contradistinction to cotton, there is only necessary in the present case to extend the duration of this treatment and also to increase the temperature.

It is also possible to add previously a suitable oxidizing agent to the padding bath. In this case, in order to develop the dyeing, a prolonged treatment with acid at increased temperature may suffice. Thus one obtains normal dyeings, without the fibre getting injured; these dyeings possess the excellent fastness properties of vat dyestuffs.

By an addition of 2–4% of diethyltartrate or like substance to the padding bath the absorption power of the fibre for the ester salt becomes substantially increased and deeper shades are obtained, without necessitating the above prolonged treatment nor an increased temperature.

According to the new process it is possible to dye not only pure cellulose acetate, but also mixed fabrics containing cellulose acetate and cotton for instance. Such a mixed fabric is padded with a solution of an ester salt, dried and steamed. Then it is developed in an oxidizing bath. Thus both kinds of fibres are almost evenly dyed. If the fabric is rinsed or soaped after the steaming, in cases where the ester salt possesses no affinity to cotton, the same is almost completely dissolved away from the cotton fibre, but not from the cellulose acetate. The result obtained after the oxidation is the presence of dyed cellulose acetate threads and undyed cotton threads. For such article, for instance the ester salt of tetrabromindigo may be used very successfully.

On the other hand it is also possible to obtain dyed cotton threads and undyed cellulose acetate threads by avoiding all that is necessary in the above process, in order to dye the cellulose acetate, namely, the steaming operation previous to the development and the prolonged treatment in the oxidizing bath at increased temperature. The fabric has only to be padded, wrung, dried and treated in the oxidizing bath for as short a period as is only required to dye the cotton normally, and without raising the temperature necessary for the development. When afterwards washed and soaped, the cellulose acetate is obtained practically white.

The above process must not be confounded with the one of German specification No. 433,146. According to the latter specification, the fibre is steamed with an ester salt and an oxidizing agent suitable for the given conditions, whereby oxidation takes place and the dyeing is obtained directly. This latter process cannot be applied to cellulose acetate, because the development proceeds too rapidly. There is not sufficient time given to the ester salt to penetrate through the fibre; the dyestuff is merely precipitated on

its surface. One obtains again dyeings which are not fast to washing and rubbing.

On the contrary, according to the present process, the cellulose acetate is steamed with the ester salt at first, without active oxidizing agent, and only after the steaming the dyeing is developed by way of oxidation.

The improved process is explained by the following examples:

1. For padding cellulose acetate use is made of a solution of 50 parts of the ester salt of tetrabromindigo in 685 parts of water, with the addition of 250 parts of a tragacanth thickening and of 15 parts of nitrite. After padding, the goods are dried and steamed for 10–15 minutes, whereupon the dyeing is developed in the ordinary sulphuric acid bath at 80° C., the duration of development being of 5 minutes. Then the goods are thoroughly rinsed and soaped for 1 minute at 80° C., again rinsed and dried. A bright, strong dyeing is thus obtained.

2. A mixed fabric of cotton and cellulose acetate is padded in the same way with a solution of the ester salt of tetrabromindigo, of 4:4′-dimethyl-6:6′-dichlorthioindigo or of dimethoxydibenzanthrone etc., dried, steamed and developed. Beautiful dyeings are thus obtained. The two kinds of fibres are dyed approximately to the same depth.

Of course the above process can also be applied for printings on cellulose acetate and on mixed fabrics. Furthermore, the process can also be applied to other cellulose esters, such as for example the cellulose formate.

In the foregoing description, I understand by vat dyestuff not only the common vat dyestuff of the indigo, thioindigo, anthraquinone, quinoneanilid-series etc., but also such coloring matters that are capable of being vatted, and especially those the leuco bodies of which do not possess any, or only a slight, affinity to fibres. With the assistance of the ester salts of such bodies, suitable dyeings are obtainable on cellulose acetate according to the present invention. Apart from anthraquinone itself, alpha-beta-naphthanthraquinone for instance (see colour index No. 1094, see also French specification 551,666) may enter into consideration. With the ester salt of leuco alpha-beta-naphthanthraquinone yellow dyeings are obtained on cellulose acetate.

What I claim is:

1. A process for dyeing and printing cellulose esters with vat dyestuffs, consisting in applying to said fibre a solution of an ester salt of a leuco vat dyestuff, drying and steaming in order to fix said ester salt as such on the fibre and then developing the dyeing by acid oxidation.

2. A process for dyeing and printing cellulose esters with vat dyestuffs, consisting in applying to a fibre of cellulose acetate a solution of an ester salt of a leuco vat dyestuff, drying and steaming in order to fix said ester salt as such on the fibre and then developing the dyeing by acid oxidation.

3. A process for dyeing and printing cellulose esters with vat dyestuffs, consisting in applying to a fibre of cellulose acetate a solution of an ester salt of a leuco vat dyestuff, drying and steaming in order to fix said ester salt as such on the fibre and then developing the dyeing by acid oxidation at a temperature of at least 70° C. and for at least 2 minutes.

4. A process for dyeing and printing cellulose esters with vat dye stuffs, consisting in applying to a fibre of cellulose acetate a solution of the ester salt of leucotetrabromindigo, drying and steaming in order to fix said ester salt as such on the fibre and developing the dyeing by acid oxidation at a temperature of at least 70° C. and for at least 2 minutes.

In witness whereof I have hereunto signed my name this 18th day of June, 1927.

GEORGES DE NIEDERHÄUSERN.