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COLORATION OF CELLULOSE ACETATE

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This invention relates to improvements in the colouration of textile yarns and fabrics composed wholly or partially of that form of cellulose acetate known as secondary or acetone soluble acetate.

In the specification of our co-pending application Serial No. 736,122, now Patent No. 2,622,958, we have described a process whereby cellulose acetate may be dyed with vat dyestuffs with little or no saponification of the cellulose acetate provided the dyestuff is converted to the leuco form by means of a reducing agent and an alkali other than a caustic alkali provided there is also present in the dyebath a swelling agent or agents for cellulose acetate.

We have now discovered and this discovery forms the basis of the present invention that cellulose acetate may be coloured either by dyeing or printing with vat dyestuffs with little or no saponification in the presence of sulphoxylic reducing agents such as, for example, sodium formaldehyde sulphoxylate, zinc formaldehyde sulphoxylate, or cyanamide sulphoxylic acid, which latter product is also known as formamidine sulphinic acid and its salts such as, for example, sodium cynanamide sulphoxylate with or without the addition of a mild alkaline substance such as sodium sulphite to act as a pH regulator and without the necessity of adding a swelling agent or agents for cellulose acetate to the dyebath or print paste such as ethyl alcohol or ethyl lactate.

When using sodium cyanamide sulphoxylate or sodium formaldehyde sulphoxylate for dyeing, the addition of ammonia enhances the dyeing effect.

When colouring by dyeing the vat dyestuff may be in either the pre-reduced or the unreduced form when the sulphoxylic reducing agent is introduced to the dyebath.

The addition of a mild alkaline substance such as sodium sulphite produces increased colour yields with a number of vat dyestuffs in dyeing and with many vat dyestuffs in printing.

By means of our new process colouration of cellulose acetate either by dyeing or by printing is not limited to vat dyestuffs of the indigoid or the thioindigoid class.

In obtaining colouration by dyeing the material may be treated in the dyebath at a temperature of 90° C. to 95° C. or alternatively it may be mechanically impregnated with the dye liquor at a temperature of 40° C. to 50° C. and subsequently steamed in saturated air free steam for 5 to 10 minutes in order to fix the colour without the necessity for any intermediate drying process. Thus the material may be impregnated with the dye liquor on a padding mangle and then passed through the steaming unit in one continuous operation and subsequently washed, given an oxidising treatment, washed, soaped, washed and dried in any convenient manner.

Alternatively the material composed wholly or partially of cellulose acetate may be mechanically impregnated with the dye liquor dried and then steamed under pressure for 5 to 10 minutes at say 15 lbs. per square inch, in which case, increased colour yields are obtained and furthermore, if the material is composed of cellulose acetate and cotton or/and viscose then colouration of the other fibre or fibres is obtained at the same time as the cellulose acetate.

In obtaining colouration by printing, after printing and drying the colours may be fixed by steaming in saturated

air free steam for 1 hour, or alternatively by steaming under pressure for 5 to 10 minutes.

The steaming pressure may vary between fairly wide limits but the nearer to atmospheric pressure the lower the colour yields which are obtained both in dyeing and printing.

We have obtained excellent results by steaming for 5 to 10 minutes at a pressure of 15 lbs. per square inch.

In printing by steaming under pressure greatly increased colour yields are obtained and in a fabric constructed of cellulose acetate and cotton or/and viscose printed with vat colours and steamed under pressure colouration of the other fibre or fibres is obtained at the same time as the cellulose acetate.

In producing colouration by dyeing the sulphoxylic reducing agents may, if desired, be produced in situ in the dye liquor. In some cases advantages accrue to this method of procedure in that increased colour yields are obtained.

Our new process is not limited to vat dyestuffs of the indigoid or thioindigoid class and we have obtained good results with a wide range of vat dyestuffs of the anthraquinone class.

We have, however, obtained unsatisfactory results with a few vat dyestuffs and it is therefor advisable before using a dyestuff to carry out a preliminary test, by any of the methods outlined in our examples to make sure that the dyestuff is suitable for use in the process.

The following examples are illustrative of the invention but the latter is in no way limited thereto:

Example 1.—Dyeing

A dyebath is made up as follows:

0.3 gramme Caledon Red BN 200% (Colour Index No. 1162)
10.0 grammes sodium formaldehyde sulphoxylate
250.0 ccs. water

The bath is vatted at 60° C. for 5 minutes. A fabric composed of 100% cellulose acetate is dyed is this bath at a temperature of 90° C. to 95° C. for 10 to 15 minutes. It is then washed, allowed to air oxidise and then soaped in a bath containing 2 grammes per litre of soap, washed and dried.

The cellulose acetate is coloured a red shade.

Example 2.—Dyeing

A dyebath is made up as follows:

0.3 gramme of Caledon Brilliant Orange 6 RS (Prototype 116)
5.0 grammes of sodium formaldehyde sulphoxylate
5.0 grammes of crystalline sodium sulphite
250 ccs. of water

The bath is vatted at 60° C. for 5 minutes. A fabric constructed of 100% cellulose acetate is dyed in this bath at a temperature of 90° C. to 95° C. for 10 to 15 minutes. It is then washed, soaped, washed and dried exactly as in Example 1.

Example 3.—Dyeing

A dyebath is made up as follows:

0.3 gramme of Caledon Brilliant Purple 4 RPS (Colour Index No. 1104)
2.0 grammes of formamidine sulphinic acid
10.0 ccs. of ammonia (sp. gr. 0.880)
2.0 grammes sodium dinaphthyl methane BB disulphonate (a dispersing agent)
250 ccs. of water

The bath is vatted at 60° C. for 5 minutes. A fabric constructed of 100% cellulose acetate is dyed in this bath for 10 to 15 minutes at a temperature of 90° C. to 95° C. It is then washed, air oxidised, soaped, washed and dried exactly as Example 1.

The material is coloured a full purple shade.

Example 4.—Dyeing

A dyebath is made up as follows:

0.3 gramme of Caledon Dark Blue GS (similar to Indanthrene Navy Blue G)

1.25 grammes of formamidine sulphinic acid
250.0 ccs. of water
2.0 grammes sodium dinaphthylmethane BB disulphonate

The bath is vatted at 60° C. for 5 minutes. A fabric constructed of 100% cellulose acetate is dyed in this bath for 10 to 15 minutes at a temperature of 90° C. to 95° C. It is then washed, air oxidised, soaped, washed and dried exactly as in Example 1.

The material is coloured a full shade of blue.

Example 5.—Dyeing

A dyebath is made up as follows:

0.3 gramme of Caledon Red Violet 2 RNS (Colour Index No. 1161)
1.25 grammes of formamidine sulphinic acid
1.3 ccs. of caustic soda 60° Tw. to convert the formamidine sulphinic acid to its sodium salt
2.0 grammes of sodium dinaphthylmethane BB disulphonate
250.0 ccs. of water

The bath is vatted at 60° C. for 5 minutes. A fabric constructed of 100% cellulose acetate is dyed in this bath for 10 to 15 minutes at a temperature of 90° C. to 95° C. It is then washed, air oxidised, soaped, washed and dried, exactly as in Example 1.

The material is coloured a red violet shade.

Example 6.—Dyeing.—Pre-reduction of the dyestuff and conversion of excess sodium hydrosulphite into sodium formaldehyde sulphoxylate, and formaldehyde bisulphite in situ

A dyebath is made up as follows:

0.3 gramme of Caledon Yellow 5 GK 150% are vatted in 220.0 ccs. of water with
2.5 ccs. of 60° Tw. caustic soda and
3.0 grammes of sodium hydrosulphite for 5 minutes at 60° C.

Then the caustic soda is neutralized by the addition of 12.5 ccs. of formaldehyde sulphurous acid (20%) solution and finally 2.5 ccs. of formaldehyde (40%) are added to convert the excess sodium hydrosulphite into formaldehyde bisulphite and sodium formaldehyde sulphoxylate.

A fabric constructed of 100% cellulose acetate is dyed in this bath for 10 to 15 minutes. It is then washed, air oxidised, soaped, washed and dried exactly as in Example 1.

The material is coloured a good yellow shade.

The addition of a further 10 grammes of sodium formaldehyde sulphoxylate to the above dyebath greatly increases the colour yields obtained.

Example 7.—Dyeing by mechanical impregnation

A padding liquor is made up as follows:

25 grammes Caledon Jade Green XN 300 (Colour Index No. 1101)
75 ccs. of 15% sodium formaldehyde sulphoxylate solution
5 grammes of 6% gum tragacanth paste in water.

A fabric constructed of cellulose acetate and cotton is mechanically impregnated with the above liquor by padding through a mangle, the pressure on the rollers being adjusted so that the material retained 80%–90% of its own weight of the liquor. After impregnation the material is dried and then steamed under pressure at 15 lbs. per square inch for 10 minutes. After steaming the material is washed in cold water, given an oxidising treatment in a bath containing 5 grammes per litre of sodium bichromate and 10 ccs. per litre of acetic acid (80%) at 20° to 25° C. for 10 minutes, washed in cold water, soaped in a bath containing 2 grammes per litre of soap for 10 minutes at a temperature of 70° C., washed and dried.

Both the acetate and the cotton are coloured a green shade.

Example 8.—Dyeing by mechanical impregnation

A padding liquor is made up as follows:

25 grammes Caledon Brilliant Purple 4 RPS are mixed with

10.5 grammes of formamidine sulphinic acid finely ground and dispersed in water and
5 grammes of 6% gum tragacanth in water incorporated therewith.

A fabric constructed of acetate and cotton is mechanically impregnated in this liquor, dried, steamed under pressure, washed, oxidised, washed, soaped, washed and dried exactly as in Example 7.

The acetate is coloured a purple shade.

Example 9.—Dyeing by mechanical impregnation

A padding liquor is made up as follows:

20 grammes of Caledon Red BN 200% paste
20 grammes of sodium formaldehyde sulphoxylate
20 grammes of sodium sulphite crystals
20 grammes of 6% gum tragacanth in water and bulked to 200 ccs. with water.

A fabric constructed of acetate and cotton is mechanically impregnated with this liquor at a temperature of 30° C. as in Example 7.

After padding and without any intermediate drying it is steamed in saturated air free steam for 3 minutes.

After steaming the material is washed in cold water, oxidised, washed, soaped, washed and dried exactly as in Example 7.

The acetate and cotton are both coloured a red shade.

Example 10.—Dyeing

A dyebath is made up as follows:

0.3 gramme of Caledon Olive Green B 200% (Prototype type 293)
230.0 ccs. of water
2.0 grammes of dinaphthylmethane BB disulphonate of soda
2.5 grammes of formamidine sulphinic acid
2.6 ccs. of caustic soda 60° Tw. to convert the formamidine sulphinic acid to its sodium salt,
10.0 ccs. of industrial 30% ammonia being finally added.

The bath is vatted for 5 minutes at 60° C. A fabric constructed of cotton and acetate is entered into this dyebath and the temperature raised to 90° C. to 95° C. After dyeing in this bath for 10 minutes the material is washed, oxidised, washed, soaped, washed and dried exactly as in Example 7.

Both the acetate and cotton are coloured a good olive green shade.

Example 11.—Dyeing

A dyebath is made up as follows:

0.3 gramme of Caledon Red Violet 2 RNS
5.0 grammes of sodium formaldehyde sulphoxylate
240 ccs. of water
10 ccs. of industrial 30% ammonia

The temperature of the bath is raised to 60° C. and a fabric constructed of acetate and cotton is entered into this dyebath and the temperature raised to 90°–95° C.

After dyeing in this bath for 10 minutes the material is washed, air oxidised, soaped, washed and dried exactly as in Example 1.

The acetate is coloured a good red violet shade.

Example 12.—Printing

A printing paste is made up as follows:

20 grammes Caledon Brilliant Blue RNS (paste) (Colour Index No. 1106)
4 grammes solution salt (benzylsulphanilate of soda)
180 grammes reduction paste (as described below)

204 bulk

Reduction paste:

2500 grammes of starch paste (1.5 lbs. per gallon of water)
1000 grammes of sodium formaldehyde sulphoxylate
1000 grammes of sodium sulphite (crystalline)
500 ccs. of water

5000 bulk

A fabric constructed of 100% acetate is printed with the above printing paste by roller machine, screen, brushing or spraying or the like method of procedure and after drying is steamed in saturated air-free steam

for 1 hour. The material is then washed, given air oxidising treatment in a bath containing 5 ccs. of hydrogen peroxide (100 volumes) for 10 minutes, washed, soaped in a bath containing 2 grammes per litre of soap for 10 minutes at a temperature of 70° C., washed and dried.

A bright blue print is obtained.

Example 13.—Printing

A printing paste is made up exactly as in Example 12 and a fabric constructed of acetate and viscose printed with this paste. After drying the material is steamed under pressure for 5 minutes at a pressure of 15 lbs. per square inch.

The material is then washed, oxidised, washed, soaped, washed and dried exactly as in Example 12.

A bright blue print is obtained and both the acetate and viscose are coloured in the printed portion of the material.

Example 14.—Printing

A printing paste is made up as follows:

25 grammes of Caledon Brilliant Purple 4 RPS (paste)
75 grammes of reduction paste (as described below)
100 bulk

Reduction paste:

29 grammes of formamidine sulphinic acid is pasted up in
50 ccs. of cold water and added to
125 grammes of 6% gum tragacanth, then
46 ccs. of water are added with stirring

250 total

A fabric constructed of acetate and viscose printed with the above printing paste, dried and then steamed under pressure (15 lbs. per square inch) for 10 minutes. It is then washed, oxidised, washed, soaped, washed and dried exactly as in Example 12.

A red print is obtained and both the acetate and viscose are coloured in the printed portion.

Example 15.—Printing

A printing paste is made up as follows:

100 grammes Caledon Yellow GN (paste) (Colour Index No. 1118)
150 grammes sodium formaldehyde sulphoxylate
750 grammes starch paste (1 lb. per gallon of water)

1000 bulk

A 100% cellulose acetate satin is printed with the above paste and after drying is steamed under pressure (15 lbs. per square inch) for 10 minutes. The material is then washed, oxidised, washed, soaped, washed and dried exactly as in Example 12.

A yellow print is obtained.

We claim:

1. Process for dyeing textile material comprising cellulose acetate which process includes the steps of immersing the material in a dyebath consisting essentially of water, a vat dyestuff, and a sulphoxylic reducing agent, at temperatures of the order of 90° C.–100° C., and for from 10 to 15 minutes, and subsequently oxidizing the dyestuff.

2. Process of claim 1, in which the reducing agent is sodium formaldehyde sulphoxylate.

3. Process of claim 1, in which the reducing agent is zinc formaldehyde sulphoxylate.

4. Process of claim 1, in which the reducing agent is cyanamide sulphoxylic acid, also known as formamidine sulphinic acid.

5. Process according to claim 1, in which the dyebath also contains an alkaline pH regulator of insufficient alkalinity to cause substantial saponification of cellulose acetate under the conditions of the process.

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