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PRINTING PROCESS FOR TEXTILES

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The present invention relates to an improved textile printing process with leuco ester salts of vat dyestuffs.

It is known that some leuco ester salts of vat dyestuffs such as Indigosol Green IB (see Schultz, Farbstofftabellen, 2d vol., page 133), Indigosol Green IGG and Olive Green IB (see Schultz, Farbstofftabellen, complemental vol. II, page 209), Indigosol Red IFBB (see Schultz, Farbstofftabellen, complemental vol. II, page 209), Indigosol Brilliant Violet 14R give unsatisfactory results in printing, when developed by one of the known steaming developing processes, especially on regenerated cellulose such as rayon crêpe, vistra mousseline, Bemberg silk and the like. Especially printings obtained with Indigosol Green IB, Indigosol Green IGG and Indigosol Olive Green IB are very poor. It is believed that this behaviour of these dyestuffs is due to their ready cleavage and to the size of the particles. The practical tests in the textile printing industry have shown that the above named Indigosol dyestuffs can satisfactorily be fixed on fibres of regenerated cellulose, when they are first subjected to a previous steaming and afterwards developed by the so-called wet-developing processes, like the nitrite process. The previous steaming makes it possible for these dyestuffs to penetrate into the fibre before they become split and for the fixation of the Indigosols to take place in the inner parts of the fibre when they become developed.

In order to obviate the taking place of an undue rapid splitting of the Indigosols during the steaming process, it is necessary to reduce in the printed pastes the quantity of the acid splitting agents, such as ammonium thiocyanate, diethyltartrate, etc., of oxidising agents such as sodium chlorate, ammonium chlorate and of catalysts such as ammonium vanadate. These changes of the printing recipe require an increase of the duration of the steaming operation from 15 minutes to about 45 to 60 minutes, otherwise the Indigosols would not be split at all. Such a long steaming process is generally not desired and especially in such cases where the fabrics have been printed not only with Indigosols, but with other dyestuffs, such as Rapidogen or Rapidecht dyestuffs, which become more or less destroyed by a long steaming operation and do not give brilliant prints. Although the steaming operation is extended, the prints obtained with the Indigosols by this method are not as deep as those obtained by the wet development processes with a previous steaming operation.

It has now been found that a too rapid splitting of the leuco ester salts of vat dyestuffs and, therefore, their bad fixation in printing on fibres from natural and regenerated cellulose can be avoided by adding to the printing pastes containing besides the leuco ester salts, a certain amount of oxidising agents, of acid splitting agents, of catalysts and of other usual additions, such organic bases that are stable towards the oxidising agents in absence of inorganic acids and by printing this paste on the fabrics and developing the dyestuffs by a short steaming. By the addition of the above cited bases a rapid and satisfactory fixation of the dyestuffs generated from the leuco esters is obtained, without any previous splitting taking place. The printings obtained in this way are much deeper than those obtained by any of the known steam-developing processes. The printing pastes containing the above said bases are further more stable than the usual printing pastes, which is naturally of great importance where easily cleavable Indigosols are used.

As organic bases that are especially suitable for carrying out the present process can be cited the aliphatic bases like mono-, di- and triethanolamine, diethylmonoethanol amine, monoethanolethylenediamine, diethylenetriamine, triethylene tetramine, as cycloaliphatic bases, for example hexahydroaniline, as aromatic bases aniline, N-ethylglycerylaniline and N-diglycerylaniline, and as heterocyclic bases pyridine, piperazine and the like. If the base employed is easily soluble in water, it can be directly added to the already prepared printing paste. In cases where the base is not easily soluble, it is preferable to dissolve it in a solvent such as ethanol, thiodiglycol and the like and to add its solution to the printing paste.

The fabrics from cellulose or regenerated cellulose printed with the printing pastes prepared as above described are after printing dried in the usual manner and steamed for 10–30 minutes, whereby a good penetration and development of the leuco esters will be obtained. The printings thus obtained do not bleed during soaping operation and give full brilliant patterns. It is obvious that the quantity of the acid splitting agent, of the oxidizing agent and of the catalysts must be adopted to the steaming equipment and steaming method used in the respective printing house. Generally it can be said, that an increase of the quantity of the acid splitting agents will reduce the time of steaming which has been increased by the addition of the base.

The prints obtained according to the present process are superior in respect of the yield of the dyestuffs to the generally known developing processes such as the sol-developing process, the

chlorate developing process, the ammonium thiocyanate-chlorate- and ammoniumchlorate steam processes and sometimes also the so-called nitrite process.

The present process further presents the great advantage of being able to be combined with the use of other dyestuffs, because the steaming operation is very short and without influence on other dyestuffs like the Rapidogen and Rapidecht dyestuffs.

The following examples, without being limitative, illustrate the present process, the parts being by weight:

Example 1

A piece of vistra mousseline is printed with Indigosol Green IB (Schultz, Farbstofftabellen, 2nd vol., page 133) with a printing paste of the following ocmposition:

	Parts
Indigosol Green IB	80
Dehagen O (German Patent 601,860) being a mixture of urea with phenols and alcohols with the boiling point superior to 100° C	100
Hot water	200
Neutral starch tragacanth thickening	500–480
Sodium chlorate solution of 25%	50
Ammonium vanadate solution of 1%	10
Diethyl tartrate	40
Organic base	20– 40
(Approx.)	1,000

The following table shows a series of organic bases that can be used and how long the printing fabric must be steamed in each case:

Organic base	Steaming period
	Minutes
20 parts of triethanolamine	25
20 parts of diethylmonoethanolamine	10
20 parts of tetraethanolammonium-hydroxide	20
10 parts of pyridine	15
20 parts of N-ethylglycerylaniline dissolved in 20 parts of alcohol	15
20 parts of N-diglycerylaniline dissolved in 20 parts of water	15
20 parts of diethylenetriamine	30
20 parts of triethylenetetramine	30
20 parts of hexahydroaniline	15
20 parts of piperazine	20
20 parts of monoethanolethylenediamine	30
20 parts of diethanolamine	25
20 parts of monoethanolamine	25
20 parts of aniline	20

The prints obtained with the above cited printing pastes are after drying steamed for 10–30 minutes, rinsed, soaped at boiling temperature, rinsed again and dried. The prints obtained in this manner are much deeper than those which are obtained by the so-called ammonium thiocyanate steaming process.

Example 2

Vistra mousseline is printed with a printing paste of the following composition:

	Parts
Indigosol Green IB (see Example 1)	80
Dehagen O (see Example 1)	100
Hot water	210
Neutral starch tragacanth thickening	500
Sodium chlorate solution of 25%	50
Ammonium vanadate solution of 1%	10
Diethyl tartrate	40
Diethylenetriamine	10
	1,000

The prints after drying are steamed for 15 to 20 minutes, soaped at the boiling temperature, rinsed and dried.

The prints obtained with the above cited printing paste are much deeper and purer than those which can be obtained by the steam developing or the wet developing process.

Example 3

A piece of vistra fabric is printed with Indigosol Green IGG, Indigosol Olive Green IB (Schultz, Farbstofftabellen, complemental vol. II, page 209) and Indigosol Red IFBB (Schultz, Farbstofftabellen, complemental vol II, page 209) in printing pastes of the following compositions:

I

	Parts
Indigosol Green IGG	80
Dehagen O (see Example 1)	100
Hot water	210
Neutral starch tragacanth thickening	500
Sodium chlorate solution of 25%	50
Ammonium vanadate solution of 1%	10
Diethyl tartrate	40
Diethylenetriamine	10
	1,000

II

	Parts
Indigosol Red IFBB	70
Thiodiglycol	50
Hot water	250
Starch tragacanth thickening	495
Diethyltartrate	50
Sodium chlorate solution 25%	55
Ammonium vanadate solution 1%	20
Diethylenetriamine	10
	1,000

III

	Parts
Indigosol Olive Green IB	60
Dehagen O (see Example 1)	100
Diethyltartrate	60
Hot water	200
Neutral starch tragacanth thickening	470
Sodium chlorate solution of 25%	80
Ammonium vanadate solution of 1%	20
Diethylenetriamine	10
	1,000

The prints obtained with the above cited pastes are after drying steamed during 15–20 minutes, rinsed, soaped at boiling temperature, rinsed again and dried. The prints obtained with these pastes are much deeper than those which can be obtained by the known steam developing process.

Example 4

A piece of Vistra-mousseline is printed with Indigosol Brilliant Violet 14R in a printing paste of the following composition:

	Parts
Indigosol Brilliant Violet 14R	50
Thiodiglycol	50
Dehagen O (see Example 1)	100
Diethyltartrate	50
Hot water	200
Neutral starch tragacanth thickening	440
Sodium chlorate solution of 25%	60
Ammonium vanadate solution of 1%	20
Triethanolamine	20
Sodium sulphoxylate-formaldehyde	10
	1,000

The prints are steamed for 10 to 15 minutes, rinsed, soaped at boiling temperature, rinsed again and dried. The print obtained according to this composition is much deeper than those which can be obtained by the well-known steam developing processes.

What I claim is:

1. A printing paste for textile of cellulosic nature, comprising an alkali metal salt of a sulfuric acid ester of a leuco-vat-dyestuff, an oxidising agent, an agent capable of splitting off acid and a catalyst and containing at least one volatile organic base which is stable towards the oxidising agent in absence of an inorganic acid, said printing paste allowing the production of deeper and fuller shades on the said textiles by a short steaming process as compared to the shades obtained by the known developing processes.

2. A printing paste for textiles of cellulosic nature, comprising an alkali metal salt of a sulfuric acid ester of a leuco-vat-dyestuff, an oxidising agent, an agent capable of splitting off acid, a catalyst and triethanolamine, said printing paste allowing the production of deeper and fuller shades on the said textiles by a short steaming process as compared to the shades obtained by the known developing processes.

3. A printing paste for textiles of cellulosic nature, comprising an alkali metal salt of a sulfuric acid ester of a leuco-vat-dyestuff, an oxidising agent, an agent capable of splitting off acid, a catalyst and diethylenetriamine, said printing paste allowing the production of deeper and fuller shades on the said textiles by a short steaming process as compared to the shades obtained by the known developing processes.

4. A printing paste for textiles of cellulosic nature, comprising an alkali metal salt of a sulfuric acid ester of a leuco-vat-dyestuff, an oxidising agent, an agent capable of splitting off acid, a catalyst and monoethanolethylenediamine, said printing paste allowing the production of deeper and fuller shades on the said textiles by a short steaming process as compared to the shades obtained by the known developing processes.

5. A process for producing deep and full shades by printing leuco ester salts of vat dyestuffs on cellulosic fabrics, comprising the step of subjecting to a short steaming cellulosic fabrics printed with a printing paste comprising an alkali metal salt of a sulfuric acid ester of a leuco-vat-dyestuff, an oxidising agent, an agent capable of splitting off acid, a catalyst and a volatile organic base which is stable to the oxidising agent in presence of an inorganic acid.

6. A process for producing deep and full shades by printing leuco ester salts of vat dyestuffs on cellulosic fabrics, comprising the step of subjecting to a short steaming cellulosic fabrics printed with a printing paste comprising an alkali metal salt of a sulfuric acid ester of a leuco-vat-dyestuff, an oxidising agent, an agent capable of splitting off acid, a catalyst and triethanolamine.

7. A process for producing deep and full shades by printing leuco ester salts of vat dyestuffs on cellulosic fabrics, comprising the step of subjecting to a short steaming cellulosic fabrics printed with a printing paste comprising an alkali metal salt of a sulfuric acid ester of a leuco-vat-dyestuff, an oxidising agent, an agent capable of splitting off acid, a catalyst and diethylenetriamine.

8. A process for producing deep and full shades by printing leuco ester salts of vat dyestuffs on cellulosic fabrics, comprising the step of subjecting to a short steaming cellulosic fabrics printed with a printing paste comprising an alkali metal salt of a sulfuric acid ester of a leuco-vat-dyestuff, an oxidising agent, an agent capable of splitting off acid, a catalyst and monoethanolethylenediamine.

ALEXIS WIAZMITINOW.