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TEXTILE PRINTING METHOD

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This invention relates to the art of ingrain textile printing, and is directed particularly to an inexpensive and simple method for the production of prints colored with azo dyestuffs.

Conventional practice in the ingrain printing of azo colors involves two techniques. In the first method, a soluble diazotized amine is introduced into a thickener dispersion to make a printing paste; the paste is printed on cloth impregnated with a coupling component, and dried to set the paste and permit handling. The coupling occurs immediately after printing; the cloth is washed out to remove the thickener of the printing paste, unreacted coupling components, etc., and is then finished.

To overcome the expense involved in preparing the impregnated fabric, and the waste involved—particularly in lightly colored patterns—in washing out of the unused coupling component, stabilized azo compounds (e. g. triazenes) have been used on non-impregnated cloth. These are mixed with alkali and coupling components, and are printed on fabrics with conventional thickeners. The printed fabric is then dried both to set the print in place, and to enable the goods to be handled. The fabric is then exposed to acid fumes in an "acid ager," where the stabilized diazo compound is hydrolyzed to yield the free diazo compound, which then couples with the coupling component. The fabric is then washed to remove thickener and unreacted intermediates, and finished in conventional fashion.

It has also been proposed (see, for example, Kircheisen U. S. Patent No. 1,646,296, issued October 18, 1927) to print on fabrics with similar pastes, using a diazotizable amine and a soluble nitrite, with or without a coupling component, depending on whether printing is done on an impregnated or plain fabric; the printed fabric is dried, acid aged and finished as usual. Such a method would obviously be far less expensive than either of the older methods, since it would save the expense of azotization of the amine in substance, and the losses attendant thereon. However, the process has never been adopted commercially, because of the fact that the color yields obtained have been so poor that the process costs more than when azotization was done in substance in accordance with conventional practice. Furthermore, the prints are less brilliant than those conventionally obtained, and tend to be more blurred.

We have discovered that excellent prints, with color yields equal to those obtained by conventional practice, may be obtained from pastes con-

taining an azotizable amine, a soluble nitrite, and preferably a coupling component, by printing the pastes in conventional fashion on a fabric, and acid ageing the fabric before the prints have been allowed to dry. If the prints are acid aged while wet, full color strength is developed, whereas for some reason drying of the prints results in considerably reduced reactivity, even though the prints are thoroughly rewet, either in the acid ager, or before entering it. Peculiarly, our prints give considerably sharper outlines than those obtained when the pastes are dried before acid ageing.

Another important factor is the state of distribution of the various components, particularly of the amine, which is of course ordinarily insoluble in the neutral or alkaline medium employed; the nitrite and coupling component are ordinarily in solution. It is important that the particles be so well distributed that the individual particles be no larger than microscopic in size (i. e. invisible to the naked eye); preferably, they are of no greater than colloidal size. This distribution may be obtained with a liquid amine by high speed stirring. With a normally solid amine, milling may be employed. Preferably, we dissolve the amine in a water-miscible organic solvent therefor, and mix this solution with the paste; with adequate stirring, the amine precipitates in colloidal-sized particles.

Obviously, our invention can be practiced most economically by including the coupling component in the printing paste; however, it can be used with impregnated fabric.

Typical examples of our invention are the following:

EXAMPLES 1 TO 5

PASTE WITH STARCH TRAGACANTH

Formulation No. 1

35.18 grams 6% gum tragacanth
are mixed with—
20.00 grams of the following:
44.8 grams wheat starch
56.0 cc. water
To this is added (hot)
.01 mol coupling component
2.50 grams sodium nitrite (as a 50% solution)
3.50 cc. 10N sodium hydroxide
32.50 cc. water (2.5 cc. from nitrite solution)

While this mix is well stirred, .01 mol amine, dissolved in 5 grams of Cellosolve (or any water-soluble organic amine solvent) are added to it. The resulting composition is agitated well, print-

ed on cotton in the conventional manner, and treated in an acid ager, whereupon the color develops within 5 seconds.

TABLE I

Ex-ample No.	Mols coupling component	Mols amine	Color
1-----	.0122M Naphthol AS—OL (ortho aniside of beta-oxy-naphthoic acid—B. O. N.).	.01M Red RC (5 chlor ortho anisidine).	Red.
2-----	.01M Naphthol AS (anilide of B. O. N.).	.005M Blue B (di-anisidine).	Blue.
3-----	.005M Naphthol AS—G (diaceto acetyl ortho toluidine).	.01M Red KB (5 chlor ortho toluidine).	Yellow.
4-----	.01M Naphthanil OP (ortho phenetidine of B. O. N.).	.01M Red KB (5 chlor ortho toluidine).	Scarlet.
5-----	.01M Naphthol AS—D (ortho toluidine of B. O. N.).	.01M Red KB (5 chlor ortho toluidine).	Red.

EXAMPLES 6 TO 10

"SUPERTEX" THICKENER

Formulation No. 2

.01 mol coupling component is pasted with—
 2.30 drops Turkey red oil
 and dissolved in—
 2.50 cc. 10N sodium hydroxide
 22.00 cc. hot water
 4.00 cc. 50% sodium nitrite solution
 is added to above. This mix is well stirred into—
 60.00 grams of a 21% "Suptertex" solution (proprietary thickener—a water-soluble gum).
 .01 mol of the amine
 is dissolved in—
 5.00 gms. of "Cellosolve" (or any organic water-soluble amine solvent)

This solution is added to the above with rapid agitation. The resulting printing composition is printed on textile fiber in the usual way, and acid aged immediately while the prints are still wet, whereupon the color develops rapidly.

TABLE II

Ex-ample No.	Mols coupling component	Mols amine	Color
6-----	.0122M Naphthol AS—OL (ortho aniside of B. O. N.).	.01M Red RC (5 chlor ortho anisidine).	Red.
7-----	.005M Naphthol AS—G (diaceto acetyl ortho toluidine).	.01M Red KB (5 chlor ortho toluidine).	Yellow.
8-----	.01M Naphthol AS (anilide of B. O. N.).	.01M Fast Yellow G (ortho chlor aniline).	Orange.
9-----	.01M Naphthanil OP (ortho phenetidine of B. O. N.).	.01M Red KB (5 chlor ortho toluidine).	Scarlet.
10-----	.01M Naphthol AS—D (ortho toluidine of B. O. N.).	.01M Red KB (5 chlor ortho toluidine).	Red.

In these examples it is noted that the solvent method of amine dispersion is again used.

Acetone, other water-soluble ketones, the "Cellosolves" (mono-alkyl ethers of ethylene glycol), the "Carbitols" (mono-alkyl ethers of diethylene glycol) and the water-miscible alcohols, can all be used to replace "Cellosolve."

EXAMPLE 11

Examples 1 and 6 were duplicated, using no water-soluble solvent. The amine was milled into the paste to obtain distribution. While good color strength was obtained, the prints lacked the full brilliance of the prints of Examples 1 and 6.

Our process eliminates the present practice of drying the cloth and rewinding after printing and before acid ageing, since the cloth goes directly into the acid ager after printing in order to get the desired color value.

Obviously, the examples are only indicative of what can be done with our process. Other thickeners can be used (e. g. water-soluble cellulose ethers, polyvinyl alcohol, alginates, starch, etc.); and the aqueous solution can be thickened by emulsification with a non-aqueous system in known manner. Furthermore, any desirable amines and coupling components can be used, in manner well known to the art.

We claim:

1. The method of printing a fabric with an in-grain azo color, which comprises laying down a wet non-acid print on the fabric from a thickened printing paste containing an azotizable amine, a soluble nitrite, and a coupling component, all in particles of no greater than microscopic size, and passing the fabric, while the print is still wet, into an acid ager, maintaining the printed fabric in the ager until diazotization and coupling has occurred, and finishing the printed fabric in conventional fashion.

2. The method of printing a fabric with an in-grain azo color, which comprises laying down a wet non-acid print on the fabric from a thickened printing paste containing an azotizable amine, a soluble nitrite, and a coupling component, all in particles of no greater than colloidal size, and passing the fabric, while the print is still wet, into an acid ager, maintaining the printed fabric in the ager until diazotization and coupling has occurred, and finishing the printed fabric in conventional fashion.

3. The method which comprises dissolving an azotizable amine in a water-miscible organic solvent, mixing the solution as an ingredient of a thickened aqueous liquid containing a nitrite and a coupling component in solution, whereby the amine is distributed through the thickened solution in particles of colloidal size, printing on a textile fabric with the thickened liquid, and passing the fabric, while the print is still wet, into an acid ager, maintaining the printed fabric in the ager until diazotization and coupling has occurred, and finishing the printed fabric in conventional fashion.

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