

UNITED STATES PATENT OFFICE

2,301,481

TEXTILE FABRIC AND METHOD OF
PREPARATION THEREOF

William W. Trowell, Greenville, S. C., assignor to
Hercules Powder Company, Wilmington, Del.,
a corporation of Delaware

No Drawing. Application July 22, 1939,
Serial No. 285,965

7 Claims. (Cl. 8—18)

This invention relates to a method of simultaneously dyeing and finishing a textile fabric, and more particularly to an improved method of producing a dyed textile fabric having a permanent finish.

One object of this invention is to provide a method for producing a permanently sized, dyed textile fabric having a superior dye fastness to washing fluids and having a finish which is resistant to laundering.

A further object of this invention is to provide a method of simultaneously dyeing and permanently finishing a textile fabric so as to provide a dye-fast, pliable, flexible, permanently finished textile from which the finish is not removed by the usual washing fluids, which is dye fast to the same washing fluids and which possesses improved color strength.

The above objects may be accomplished in accordance with this invention by impregnating a textile fabric with an aqueous emulsion comprising a water-immiscible solution of a water-insoluble ethyl cellulose, the said emulsion containing in the water phase a direct, water-soluble dye-stuff, removing the excess emulsion from the fabric, evaporating the water and volatile solvent from the impregnated fabric, and pressing the fabric at an elevated temperature.

In carrying out the method in accordance with this invention, a textile fabric is impregnated with an aqueous emulsion of a solution of water-insoluble ethyl cellulose in a volatile, substantially water-immiscible solvent, the said aqueous emulsion containing in the water phase a direct, water-soluble dyestuff. A plasticizer for the ethyl cellulose which is substantially inert to the action of aqueous alkaline washing solution and also a synthetic resin of suitable type may be included in the emulsion if desired.

In following my method, the excess emulsion may be conveniently removed from the fabric by passing the cloth between padded squeeze rolls. The fabric is then preferably dried at an elevated temperature to remove the water and volatile solvent from the impregnated fabric and then pressed or ironed preferably at a temperature within the range of about 180° F. to about 400° F., and more preferably within the range of about 240° F. to about 280° F., the actual temperature used being dependent upon the type of fabric employed and the particular composition deposited on the fabric. The ironing process may be accomplished by passing the treated fabric over a series of heated rolls or by drying the fabric in a

blast of heated air and subsequently passing it between heated pressure rolls.

The amount of emulsion remaining in the fabric after removal of the excess by passing between the padded squeeze rolls will determine to a large extent the final stiffness, hand and body of the finished dyed fabric. By impregnating the fabrics with emulsions containing different concentrations of ethyl cellulose with or without the plasticizers and synthetic resins it is possible to vary the amount of solids deposited in the fabric and thereby to vary the hand and body of the finished fabric as desired. In any event, the amount of emulsion remaining in the fabric after the excess emulsion has been removed will be such that the amount of dry, film-forming composition remaining in the fabric will, at the end of my procedure, be insufficient to fill the interstices between threads or to form a continuous coating over the surface of the fabric, but will be sufficient to provide the finished fabric herein described.

In practicing my invention, I apply my ethyl cellulose emulsions containing a direct water-soluble dye in the water phase so as to leave in the finished dyed fabric an amount of the thermoplastic substance in the dry state not over about 40% of the weight of the untreated fabric, and preferably not over about 20% of the weight of the untreated fabric. The amount of dry thermoplastic substance retained in the fabric is controlled depending on whether a soft, flexible, permanent finish on the one hand or a stiff, starched-like finish on the other hand, or any finish intermediate thereto is desired.

The ethyl cellulose used in the aqueous emulsions of this invention will be sufficiently etherified to be insoluble in water and inorganic solvents and will be recognized as water-insoluble ethyl cellulose. It may have, for example, an ethoxy content in the range of about 43% to about 51%, and preferably from about 44% to about 48%. The viscosity characteristics of the ethyl cellulose used will depend upon the properties desired in the ultimate composition.

The aqueous emulsion of water-insoluble ethyl cellulose used in accordance with my invention may, as stated above, contain a plasticizer for lowering the softening point of the ethyl cellulose. The plasticizer used will preferably be one which is resistant to alkaline solutions. It will also desirably be compatible with ethyl cellulose. The plasticizer may be either of the liquid type or the solid type. Solid plasticizers are preferred since

they contribute to the fullness and body of the finished fabric more than the liquid plasticizers. Suitable plasticizers are hydrogenated methyl abietate; hydrogenated ethyl abietate; triphenyl phosphate; tricresyl phosphate; tributyl phosphate; methyl phthalyl methyl glycolate; ethyl phthalyl ethyl glycolate; di-(para-tertiary butyl phenyl), mono-(5-tertiary butyl-2 xenyl) phosphate, etc.

The aqueous emulsion may also contain a suitable synthetic resin. Resins which I have found to be suitable in my invention are thermoplastic, alkali-resistant synthetic resins compatible with ethyl cellulose. Thus, I have found that I may use rosin-modified alkyd resins, such as rosin modified or hydrogenated rosin modified terpene-maleic anhydride resins, rosin modified maleate resins; drying oil modified terpene-maleic anhydride resins; polymerized hydrocarbon resins, such as polymerized naphthalene, polymerized coumarone-indene or hydrogenated forms thereof; 100% phenolic resins; polyhydric alcohol esters of hydrogenated rosin, as well as resins of other types which are of a thermoplastic character, are alkali-resistant and are compatible with ethyl cellulose. Where optimum results are desirable, I prefer to employ resins falling within the above classification which are light in color and which have a relatively low acid number.

The aqueous ethyl cellulose emulsions used in accordance with this invention will preferably be prepared by emulsifying in water a water-immiscible solution of water-insoluble ethyl cellulose or of water-insoluble ethyl cellulose mixed with either a plasticizer or a synthetic resin, or both, in a suitable common solvent therefor, preferably a mixed solvent comprising light aromatic and petroleum hydrocarbons and butanol. The emulsions will be of the oil-in-water type. Suitable emulsifying agents are, for example, the alkali metal salts of the higher aliphatic half ester sulfates, as sodium lauryl sulfate, sodium stearyl sulfate, etc.; sulfonated oils, as sulfonated castor oil, sulfonated olive oil, etc.; soaps, as sodium stearate, potassium oleate; sodium oleate, etc.; alkali metal salts of alkyl naphthalene sulfonic acids, as sodium butyl naphthalene sulfonate, sodium isopropyl naphthalene sulfonate, etc.

In the water phase of the ethyl cellulose emulsions which I employ, a direct water-soluble dyestuff will be present. Dyestuffs belonging to this class are, for example:

	Color Index No.
Pontamine Fast Red 8BL.....	278
Pontamine Fast Green GL.....	None
Pontamine Yellow CH.....	365
Pontamine Sky Blue FF.....	518
Pontamine Fast Brown RKL.....	None
Pontamine Fast Orange.....	Pr 56
Pontamine Black E.....	581
Pontamine Blue BBN Conc.....	406
Pontamine Brown BT Conc.....	561
Pontamine Brown RMR.....	420
Pontamine Fast Black FF.....	539
Pontamine Fast Blue RRL.....	319
Pontamine Fast Brown BRL.....	Pr 41
Pontamine Fast Orange ERL.....	Pr 56
Pontamine Fast Orange MRL.....	621
Pontamine Fast Orange S.....	326
Pontamine Fast Red F.....	419
Pontamine Fast Scarlet 4BA.....	326
Pontamine Fast Yellow 4GL Conc.....	Pr 43
Pontamine Yellow>NNL.....	814

The direct water-soluble dyestuff will be present

in the emulsion in an amount not exceeding about 0.5% by weight of the total emulsion. It may preferably be included in the water used to dilute the concentrated emulsion.

My improved process of simultaneously finishing and dyeing textile fabrics is applicable to any of the textile materials such as for example, organdy, lace, broadcloth, cotton, wool, rayon, etc. Naturally, due to the different qualities of fabric to be treated, their weight, their degree of absorbency, etc., it is impossible to define the amount of impregnating composition required to provide either a soft, flexible finish or a stiff, starched-like finish. Generally speaking, however, a maximum of about 40% by weight of solid thermoplastic substance in the fabric, based on the weight of the untreated fabric, is contemplated. For most purposes, not over about 20% by weight is contemplated.

The concentration of the solid ingredients of the emulsions used in my invention may be varied over wide ranges merely by diluting concentrated emulsions with water. By adjusting the water content of emulsions in this manner, I am enabled to very effectively control the amount of solid thermoplastic substance deposited in the fabric, and thereby to control stiffness of the finished fabric as desired.

Example I

The following solutions were prepared:

	Parts by weight
(a) Ethyl cellulose.....	32
Xylol.....	122
Hi flash naphtha.....	26
Butanol.....	20
Total.....	200
(b) Potassium oleate.....	1.5
Water.....	98.5
Total.....	100

Two hundred parts by weight of solution (a) were pre-mixed with 100 parts by weight of solution (b) by pouring solution (a) into the water phase while agitating the latter and emulsified together by passing the pre-mixed emulsion twice through a Manton-Gaulin two-stage homogenizer using about 2,000 pounds pressure on the first stage and about 2,500 pounds pressure on the second stage so that a particle size of about 5 microns to about 50 microns was obtained. Five hundred parts by weight of the emulsion were then diluted with 1,000 parts by weight of water containing 3.75 parts by weight of Pontamine fast red 8BL. The dyestuff was incorporated in the water by boiling the dye in about 1/2 of the water used in the dilution and adding the remainder of the water so as to cool the dye solution to approximately room temperature before addition to the concentrated emulsion. The diluted emulsion containing the dyestuff so obtained was then used to simultaneously impregnate and dye 2.85 yard sheeting. The excess emulsion was squeezed from the fabric and the fabric dried over dry cans at a temperature of about 275° F. The dyed and finished fabric obtained possessed a full-bodied, semi-stiff finish and had a stiffness of 33 mg. as measured on a Gurley stiffness tester. The fabric possessed improved dye strength and more level dyeing over that obtained without the use of the ethyl cellulose emulsion.

Example II

The following solutions were prepared:

	Parts by weight
(a) Ethyl cellulose.....	32
Triphenyl phosphate.....	20
Xylol.....	108
Hi flash naphtha.....	22
Butanol.....	18
Total.....	200
(b) Potassium oleate.....	1.5
Water.....	98.5
Total.....	100

Solutions (a) and (b) were emulsified as in Example I. The concentrated emulsion was diluted with a dye solution containing Pontamine fast red 8BL as in Example I, and the diluted emulsion containing the dyestuff used to simultaneously dye and finish 2.85 yard sheeting as in Example I. The resulting fabric had a stiffness of 39.6 mg. The dyed fabric obtained was resistant to washing and retained its stiffness after washing and ironing.

Example III

The following solutions were prepared:

	Parts by weight
(a) Ethyl cellulose.....	32
Triphenyl phosphate.....	6
Glycerol ester of hydrogenated rosin.....	20
Xylol.....	106
Hi flash naphtha.....	20
Butanol.....	16
Total.....	200
(b) Potassium oleate.....	1.5
Water.....	98.5
Total.....	100

Solutions (a) and (b) were emulsified as in Example I. The simultaneous dyeing and finishing procedure of Example I was repeated. The fabric obtained possessed a stiff hand having a stiffness of 90.2 mg. The dyed strength was greater than obtained without the ethyl cellulose emulsion and the fabric retained its dye and finish on laundering.

Example IV

Five hundred parts by weight of the concentrated ethyl cellulose emulsion used in Example III were diluted with 1,000 parts by weight of water containing 3.75 parts by weight of Pontamine yellow CH. The diluted emulsion contained 0.25% dye. It was used to impregnate 2.85 yard sheeting by the procedure described in Example I. The resulting fabric was full-bodied and stiff, having a stiffness of 95 mg. The dyeing was more level and the dyed strength greater than obtainable without the ethyl cellulose emulsion. The fabric also showed superior dye fastness and retained its stiffness after laundering.

Example V

Five hundred parts by weight of the concentrated ethyl cellulose emulsion used in Example III were diluted with 1,000 parts by weight of water containing 3.75 parts by weight of Pontamine sky blue FF. The mixture contained 0.25% of the dye. The diluted emulsion was then used to simultaneously dye and impregnate 2.85 yard sheeting by the procedure described in

Example I. The resulting fabric had a full, stiff finish, the stiffness being 96.3 mg. as measured on the Gurley stiffness tester. The dyed fabric possessed greater depth of shade and more level dyeing than obtainable without the ethyl cellulose emulsion. The dyed fabric retained its color and stiffness after laundering.

Example VI

Five hundred parts by weight of the concentrated emulsion used in Example III were diluted with 1,000 parts by weight of water containing 3.75 parts by weight of Pontamine fast green GL (DuPont). The mixture contained 0.25% dye. The diluted emulsion was used to simultaneously impregnate and dye 2.85 yard sheeting by the procedure described in Example I. The dyed fabric obtained was full and stiff, having a stiffness of 88.3 mg. The dyed color was stronger than obtainable without use of the ethyl cellulose emulsion and showed greater retention of color after washing.

Example VII

The following solutions were prepared:

	Parts by weight
(a) Ethyl cellulose.....	32
Triphenyl phosphate.....	16
Glycerol ester of hydrogenated rosin.....	10
Xylol.....	106
Hi flash naphtha.....	20
Butanol.....	16
Total.....	200
(b) Potassium oleate.....	1.5
Water.....	98.5
Total.....	100

Solutions (a) and (b) were emulsified as in Example I.

Five hundred parts by weight of the concentrated emulsion were then diluted with 1,000 parts by weight of water containing 3.75 parts by weight of Pontamine fast red 8BL. The dyestuff was incorporated in the water phase by the procedure described in Example I. The diluted emulsion containing the dyestuff was used to simultaneously impregnate and dye 2.85 yard sheeting using the procedure described in Example I. A stiff and full-bodied, dyed fabric was obtained having a stiffness of 69 mg., as measured on the Gurley Stiffness tester. The fabric showed an improved dyed shade over that obtainable without the ethyl cellulose emulsion. The fabric was dyed fast and retained its stiffness after laundering.

Example VIII

The following solutions were prepared:

	Parts by weight
(a) Ethyl cellulose.....	32
Hydrogenated naphthalene polymer, M. P. 130° C.....	10
Triphenyl phosphate.....	16
Xylol.....	106
Hi flash naphtha.....	20
Butanol.....	16
Total.....	200
(b) Potassium oleate.....	1.5
Water.....	98.5
Total.....	100

Solutions (a) and (b) were emulsified as in Example I.

Five hundred parts by weight of the concentrated emulsion were diluted with 1,000 parts by weight of water containing 3.75 parts by weight of Pontamine fast red 8BL. The diluted emulsion containing 0.25% by weight of the dyestuff was then used to simultaneously impregnate and dye 2.85 yard sheeting by the procedure described in Example I. The dyed fabric obtained was full-bodied and stiff, having a stiffness of 56 m. The dyed shade and the stiffness of the fabric were retained after washing.

The method of simultaneously dyeing and finishing textile fabrics in accordance with this invention enables one to obtain in a single operation a dyed and finished fabric. By simultaneously dyeing and finishing a textile fabric with an aqueous emulsion of a water-immiscible solution of water-insoluble ethyl cellulose containing in the water phase a direct water-soluble dyestuff, a dyed and finished textile fabric of improved properties is obtained, in accordance with this invention. The strength of the dyed shade and the levelness of the dyed fabric are superior to that obtainable by dyeing the fabric with a water solution of the dye, as in common practice.

It will be understood that the details and examples hereinbefore set forth are illustrative only and that the invention as herein broadly described and claimed is in no way limited thereby.

This application forms a continuation-in-part of my application Serial No. 128,039 filed February 26, 1937 entitled "Textile Fabrics and Methods of Preparation Thereof".

What I claim and desire to protect by Letters Patent is:

1. Process of simultaneously finishing and dyeing a textile fabric to provide in one operation a permanently finished, dyed fabric of open texture which comprises immersing a textile fabric in an aqueous emulsion of a water-immiscible solution of water-insoluble ethyl cellulose, said aqueous emulsion containing in the aqueous, continuous phase a water-soluble direct dye, removing the excess emulsion from the fabric so as to leave in the fabric, on drying, an amount of solids insufficient to fill the interstices between the threads or to form a continuous coating over the surface of the fabric, drying the fabric, and ironing the fabric.

2. Process of simultaneously finishing and dyeing a textile fabric to provide in one operation a permanently finished, dyed fabric of open texture which comprises immersing a textile fabric in an aqueous emulsion of a water-immiscible solution of water-insoluble ethyl cellulose and a plasticizer therefor which is resistant to alkaline solutions, said aqueous emulsion containing in the aqueous, continuous phase a water-soluble direct dye, removing the excess emulsion from the fabric so as to leave in the fabric, on drying, an amount of solids insufficient to fill the interstices between the threads or to form a continuous coating over the surface of the fabric, drying the fabric, and ironing the fabric.

3. Process of simultaneously finishing and dyeing a textile fabric to provide in one operation a permanently finished, dyed fabric of open texture which comprises immersing a textile fabric in an aqueous emulsion of a water-immiscible solution of water-insoluble ethyl cellulose and a plasticizer therefor which is resistant to alkaline solutions and a thermoplastic alkali-resistant synthetic resin compatible with ethyl cellulose, said aqueous emulsion containing in the aqueous,

continuous phase a water-soluble direct dye, removing the excess emulsion from the fabric so as to leave in the fabric, on drying, an amount of solids insufficient to fill the interstices between the threads or to form a continuous coating over the surface of the fabric, drying the fabric, and ironing the fabric.

4. Process of simultaneously finishing and dyeing a textile fabric to provide in one operation a permanently finished, dyed fabric of open texture which comprises immersing a textile fabric in an aqueous emulsion of a water-immiscible solution of water-insoluble ethyl cellulose and a plasticizer therefor which is resistant to alkaline solutions and a thermoplastic alkali-resistant polymerized hydrocarbon resin compatible with ethyl cellulose, said aqueous emulsion containing in the aqueous, continuous phase a water-soluble direct dye, removing the excess emulsion from the fabric so as to leave in the fabric, on drying, an amount of solids insufficient to fill the interstices between the threads or to form a continuous coating over the surface of the fabric, drying the fabric, and ironing the fabric.

5. Process of simultaneously finishing and dyeing a textile fabric to provide in one operation a permanently finished, dyed fabric of open texture which comprises immersing a textile fabric in an aqueous emulsion of a water-immiscible solution of water-insoluble ethyl cellulose and a plasticizer therefor which is resistant to alkaline solutions and a thermoplastic alkali-resistant rosin-modified alkyd resin compatible with ethyl cellulose, said aqueous emulsion containing in the aqueous, continuous phase a water-soluble direct dye, removing the excess emulsion from the fabric so as to leave in the fabric, on drying, an amount of solids insufficient to fill the interstices between the threads or to form a continuous coating over the surface of the fabric, drying the fabric, and ironing the fabric.

6. Process of simultaneously finishing and dyeing a textile fabric to provide in one operation a permanently finished, dyed fabric of open texture which comprises immersing a textile fabric in an aqueous emulsion of a water-immiscible solution of water-insoluble ethyl cellulose and a plasticizer therefor which is resistant to alkaline solutions and a polyhydric alcohol ester of hydrogenated rosin, said aqueous emulsion containing in the aqueous, continuous phase a water-soluble direct dye, removing the excess emulsion from the fabric so as to leave in the fabric, on drying, an amount of solids insufficient to fill the interstices between the threads or to form a continuous coating over the surface of the fabric, drying the fabric, and ironing the fabric.

7. Process of simultaneously finishing and dyeing a textile fabric to provide in one operation a permanently finished, dyed fabric of open texture which comprises immersing a textile fabric in an aqueous emulsion of a water-immiscible solution of water-insoluble ethyl cellulose, said aqueous emulsion containing in the aqueous, continuous phase a water-soluble direct dye, removing the excess emulsion from the fabric so as to leave in the fabric, on drying, an amount of solids insufficient to fill the interstices between the threads or to form a continuous coating over the surface of the fabric, drying the fabric, and ironing the fabric at a temperature within the range of about 180° to about 400° F.

WILLIAM W. TROWELL.