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## UNITED STATES PATENT OFFICE

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## METHOD OF COLORING TEXTILES

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## 5 Claims. (Cl. 117-121)

This invention relates to the dyeing of textile fabrics, and has particular reference to a new and novel method of dyeing textile with pigment lacquers emulsified in water, and to the dye baths used in the method. Specifically, this invention refers to the use as textile dye baths of lacquer emulsions of low binder and pigment content, but containing at least as much lacquer as water by volume.

Textile fabrics are almost universally colored by being treated with a solution of a dyestuff in an aqueous medium; the soluble dyestuff is precipitated in the capillaries of the fibers of the yarns, or is caused to adhere chemically to the textile material. In some cases, water-insoluble dyestuffs having an affinity for certain synthetic fabrics are used in solution in other solvents. Since all of these dyestuffs must be capable of conversion from the soluble state to the insoluble state in contact with the fiber, and since they must be resistant to a great variety of deteriorating influences, the problem of inexpensive, fast textile colors has always been a pressing one.

The ready availability and low cost of the insoluble pigment colors has prompted many investigators to study the application of these colors for the dyeing of textiles, but with relatively little success. Except where the pigment is incorporated into a spinning solution used for making synthetic fibers, unaided mechanical adhesion of the pigment to the yarns has proven unsuccessful. Attempts to cause the pigment to adhere to the fabric by the use of binders have likewise proven abortive, for a variety of reasons. A principal cause has been the failure of the pigment binders to withstand the severe conditions to which textile are subjected; the failure of the binder leaves the pigment in mere mechanical contact with the fabric, whence it is easily removed. A second important difficulty has been that of even application of very small quantities of pigment plus binder, so that the fabric is colored uniformly. The problem is presented by the tendency of pigments to flocculate 45 or settle out in highly disperse media, so that uniform application is very difficult.

It has been proposed to dye textile fabrics with pigmented lacquers emulsified as the dission, using heat-convertible synthetic resins as binder in the lacquer. Such emulsions give satisfactory uniform dyeings on fabrics, when the fabrics are immersed in the bath and the excess heated after the dyeing operation, both to evaporate the water and solvent, and to set the resin. If the fabric is not maintained in a uniform state of tension, or if the drying is done unevenly, the color will tend to migrate and give a streaked

appearance. This tendency is considerably aggravated as cloth, thickness increases, so that shirting broadcloths and heavier fabrics must be handled with special equipment and with such extreme care that rapid drying is impossible. As a result, the emulsion pad dyeing has for the most part been restricted to rather thin fabrics, or to special cases with heavier fabrics.

I have discovered that cloth may be dyed with emulsions of pigmented lacquers in water, while substantially eliminating the tendency of the lacquer to migrate, by dyeing with a bath in which the disperse lacquer phase constitutes at least 50% of the volume of the bath.

My new method and dye baths may be used 15 successfully with any sort of fabric, and no noticeable migration occurs even on heavy fabrics such as jeans, when the fabrics are dried in conventional fashion by passing the cloth

 $_{20}$  through the ordinary driers used in textile plants. Substantially any combination of pigments and binder may be used for very cheap fabrics such as are used for temporary displays, where light-fastness, washability, resistance to dry cleaning fluids are not essential characteristics, 25or where the stiffness imparted by the pigment binder is not objectionable. In the dyeing of better grade fabrics, however, where these factors are of importance, the pigments and bind-30 ers must be chosen with care. The pigments must be light-fast, non-bleeding in dry cleaning solvents (petroleum hydrocarbons, carbon tetrachloride, etc.) and resistant to soap, while the binders must also be resistant to the same disin-35 tegrating influences, and at the same time bind the pigment to the fabrics even when only small amounts of resin are present on the fabric. Heat-convertible synthetic resins are particularly useful, including the urea formaldehyde 40 resins (such as are made by reacting formaldehyde with urea, thiourea, melamine and other homologues and derivatives of urea), the short oil alkyds, and the heat-convertible phenolic resins.

Where a heavy finish is undesirable on the fabric, the binder content added to the fabric should not exceed about  $2\frac{1}{2}\%$ , and since the fabric retains about an equal weight of dyebath, the binder content of the dyebath should not in such continuous phase of a lacquer-in-water emul- 50 cases exceed  $2\frac{1}{2}$ %. In such cases, and even when higher binder concentrations are used, the lacquer becomes so thin in body (particularly when a heat-convertible resin is the binder) that the pigment tends to flocculate and settle out. I color squeezed off. However, the cloth must be 55 have found that this difficulty can be overcome by preparing an original emulsion of a pigmented lacquer in which the pigment does not flocculate, and then reducing the total binder content of the lacquer by adding an organic phase to the emulsion containing very little or no binder, with 60

or without water. The added organic phase in such case consists essentially of a volatile solvent having dissolved therein an emulsifying agent, the solvent being of such a character that it is only partially miscible with the organic solvent, so that a three-phase emulsion is obtained. Such an emulsion is described and claimed in my co-pending application, Serial No. 351,206, filed August 3, 1940.

It consists essentially of an outer water phase 10 having two dispersed lacquer phases, one of which is a pigmented resin solvent aggregate, and the other of which consists essentially of a volatile solvent having dissolved therein sufficient emulsifying agent to make the emulsion stable, but 15 under 10% non-volatile based on the weight of this phase by preference.

Typical examples of my invention are the following:

## EXAMPLE 1

#### Pigment dyeing emulsions

	ts by	
we	lght	
Carbon blackare dispersed in—	5.0	25
Alkyd resin solution (65 resin, 35 xylene) and	10.0	
Xylene To the dispersion is added—	10.0	
Solvent-soluble urea formaldehyde resin		30
solution (50 resin, 30 butanol, 20 xylene) _	2.0	
Alkyd resin solution (50 resin, 50 toluene)	5.0	
Pine oil	10.0	
Xylene	7.0	
The lacquer is then emulsified into-		35
Water	46.0	
Sodium laury sulfate	2.0	
Dry bentonite	3.0	

The alkyd resin of this lacquer is glycerol phthalate, modified with 33% soya oil fatty acids; it can be set by heat. It is soluble in aromatic hydrocarbons, and will precipitate on further dilution with aliphatic hydrocarbons, or with mixtures containing substantial quantities of aliphatics. The tendency of this lacquer toward immiscibility with added solvents is increased by pigmentation, as with all resin.

This emulsion contains about 10% resin solids; if applied to cloth direct, it will produce too stiff a hand. Reduction of the lacquer with solvent before emulsification produces pigment flocculation. It is possible to increase the non-aqueous 55 phase and reduce the solids content by emulsifying the proper solvent into the emulsion with an emulsifying agent. This may be done by making an oil-in-water emulsion from-

	eight
enated petroleum sol-	
ge 135–177° C.)	69.00

vent-boiling range 135-177° C.)	69.00
Oleic acid	1.25
Triethanolamine	0.40
Water	29.35

Solvesso #2 (hydrog

#### 100.00

An excellent pigment dyeing bath is made from 1 part of the pigmented emulsion and 4 or more 70 parts of the unpigmented material.

If desired, the same effect may be obtained by adding water to the pigmented emulsion, then adding the emulsifying agent to the water, and then adding the solvent.

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The cloth, after impregnation with the pigment dyeing bath, is heated to remove the water and volatile solvent, and to convert the resin to the insoluble state.

## EXAMPLE 2

#### Yellow emulsion

10	Parts by w A 20% aqueous pulp of yellow pigment, pre- pared by diazotizing dichlorbenzidine and coupling with acetoacetanilide A 35% aqueous solution of sulfonated	6.5
15	Alkyl resin solution of Example 1	.25 8.5 10.0
20	The resultant water-in-lacquer emulsion is diluted with— Solvesso #2 and added slowly, on a high speed mixer, to a solution of— Sodium lauryl sulfate	48.0 2.0
<b>25</b> 30	Water The emulsion inverts as it enters the w producing a stable lacquer-in-water emu having 1.3% pigment solids, and 5.5% resin s This emulsion is then blended with equal by volume of a clear emulsion made as follows	24.75 vater, ilsion olids. parts
50	Example 3 Clear emulsion	
35	50 parts by weight of a lacquer consisting Parts by w Urea formaldehyde resin solution of Ex- ample 1	
	Alkya resin of Example 1, 50% solution in	_

100

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White pine oil Solvesso #2\_\_\_\_\_ 55 are added slowly, with stirring to a solution of-

pine oil\_\_\_\_\_

Sodium lauryl sulfate 2.545 Water\_\_\_\_\_ ..... 47.5

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to produce a stable clear lacquer-in-water emulsion containing 10% resin solids.

For the 2.5 parts of sodium lauryl sulfate, 1.5 50 parts of water and 1.5 parts of Aerosol O. T. (sodium salt of alkyl ester of sulfo succinic acid) may be used.

When applied alone, too much hand is obtained. When diluted with water, migration occurs. When diluted as suggested in Example 1,

the resultant fabric is soft, and no migration occurs on drying.

Obviously the examples may be multiplied indefinitely without departing from the scope of Parts by 60 my invention, which is defined in the claims. Obviously, while I have shown only preferred binders in my examples, natural resins, cellulose esters and ethers, natural and synthetic rubber, and other pigment-binding substances may be <sup>65</sup> applied to cloth in the indicated fashion, while avoiding migration. While my method of diluting the emulsions to obtain a low solid content is highly desirable, especially with heat-convertible resins, it is not essential where the pigment may be dispersed without flocculation otherwise, as with high viscosity cellulose derivatives.

I claim:

1. The method of pigment dyeing a fabric while avoiding substantial migration of color on drying, 78 which comprises saturating the fabric with a pigmented lacquer-in-water emulsion, the nonaqueous phase of which comprises at least 50% of the total volume, and drying the fabric.

2. The method of pigment dyeing a fabric while avoiding substantial migration of color on drying, 5 which comprises saturating the fabric with a pigmented lacquer-in-water emulsion, the nonaqueous phase of which comprises at least 50%of the total volume, the emulsion containing a heat-convertible synthetic resin to bind the pigment to the fabric, and thereafter heating the fabric to dry it and convert the resin to its insoluble state.

3. The method of pigment dyeing a fabric while avoiding substantial migration of color on 15 drying, which comprises saturating the fabric with a pigmented lacquer-in-water emulsion, the non-aqueous phase of which comprises at least 50% of the total volume, the emulsion containing a urea formaldehyde resin to bind the pig- 20

ment to the fabric, and thereafter heating the fabric to dry it and convert the resin to its insoluble state.

4. The method of claim 1 in which the binder content of the lacquer is under about  $2\frac{1}{2}\%$  of the emulsion weight.

5. The method of pigment dyeing a fabric while avoiding substantial migration of color on drying, which comprises saturating the fabric with a pigmented lacquer-in-water emulsion containing under about  $2\frac{1}{2}$ % of binder for the pigment, the non-aqueous phase comprising at least 50% of the total volume and consisting of a pigmented phase containing solvent and heat-convertible resin, and a separate phase consisting essentially of solvent having dissolved therein an agent capable of producing a stable emulsion, and thereafter heating the fabric to dry it and convert the resin to its insoluble state.

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