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2,926,987

## PREPARATION OF PHENYLPHENOL DYEING ASSISTANT COMPOSITIONS

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No Drawing. Application April 30, 1956  
Serial No. 581,309

4 Claims. (Cl. 8—93)

This invention relates to improvements in the dyeing of textile structures such as yarns, filaments, fibers and fabrics having a basis of materials that are not readily permeable to water. More particularly, the instant invention relates to an improved process for dyeing yarns, filaments, fibers and fabrics made of or containing materials which are not readily permeable to water such as, for example, the polyesters of terephthalic acid and ethylene glycol, the polymers of acrylonitrile, the copolymers of acrylonitrile and vinyl chloride, the vinyl resins, the cellulose acetates and nylon, which materials will hereinafter be designated as water-impermeable polymeric materials.

It is well known in the art of dyeing that when it is attempted to dye textile structures of water-impermeable polymeric materials by the dyeing procedures ordinarily used in dyeing textile structures of cotton, silk, wool and regenerated cellulose, the dyestuff either does not penetrate the water-impermeable polymeric materials thus effecting no coloration on said materials, or when a coloration is effected it is "fugitive" and leaches out during a washing step. It is also well known in the dyeing art that the dyeing of textile structures of water-impermeable polymeric materials may be improved by the use in the dyebath of dyeing assistants which may function as imbibing agents, swelling agents or carriers.

Among the dyeing assistants heretofore employed in the dyeing of textile structures of water-impermeable polymeric materials with acid and acetate dyestuffs are the phenylphenols, substituted and unsubstituted. The most useful of the phenylphenols and therefore most widely employed as a dyeing assistant in the dyeing of water-impermeable polymeric materials are orthophenylphenol and paraphenylphenol. However, in the prior processes using these dyeing assistants, it was necessary to predissolve said phenylphenols in a caustic solution and then to release the phenylphenol in the presence of a dispersing agent by the addition to said solution of acetic acid.

It is an important object of this invention to provide an improved process for dyeing textile structures of water-impermeable polymeric materials wherein the necessity of first forming the sodium salt of the phenylphenol and then regenerating the phenylphenol by the addition of an acid is eliminated.

Another object of this invention is the provision of a novel dyeing assistant composition containing orthophenylphenol or paraphenylphenol for use in the dyeing of textile structures of water-impermeable polymeric materials.

A further object of this invention is the provision of a novel process for the preparation of the orthophenylphenol or paraphenylphenol dyeing assistant composition.

Other objects and advantages of this invention will appear in the following detailed description and appended claims.

In accordance with the instant invention, there is employed as a dyeing assistant in the dyeing of textile

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structure of water-impermeable polymeric materials a dispersed powder of orthophenylphenol or paraphenylphenol which is prepared by working a mixture of the phenylphenol and a suitable dispersing agent in a dispersion mill in the presence of a small quantity of water, drying the resultant powder at an elevated temperature until substantially all of the water is removed, and grinding the dry powder to greater fineness in a hammer mill.

Any suitable anionic dispersing agent may be employed in the preparation of the dispersed powder of orthophenylphenol or paraphenylphenol such as, for example, sodium salts of naphthalene sulfonic acid-formaldehyde condensation products (Tamol NNO), sodium lignin sulfonates (Marasperse B and N), sodium salts of partially desulfonated lignin sulfonic acids (Marasperse CB), and secondary licorice extracts (Flotite). It is preferred, however, to use the sodium lignin sulfonate dispersing agents since they are not only effective surfactants but they are also relatively inexpensive. The amount of dispersing agent necessary to disperse the paraphenylphenol may vary from 25% to 75%.

The dyeing of the textile structures is effected in accordance with this invention by first forming a paste of the acid or acetate dyestuff and the dispersed powder of orthophenylphenol or paraphenylphenol. A dyebath is then formed from the paste in a dye vessel, and the textile structure is entered into the dyebath. The temperature of the contents of the dye vessel is raised to the boil and maintained at the boil for the required length of time after which the dyed textile structure is rinsed in cold water and dried.

The instant invention will be understood more clearly from the illustrative examples. Parts, proportions and percentages are by weight unless otherwise indicated.

### Example I

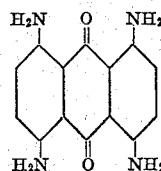
Ten parts of paraphenylphenol and 10 parts of Tamol NNO were placed in a Werner Pfleiderer type dispersion mill. Ten parts of water were added and the material was milled in a viscous state for two and one-half hours. Steam was passed through the jacket to remove part of the water to increase the viscosity of the mix during the first 15–20 minutes. The steam was then shut off. At the end of the milling period the heat of friction of milling removed most of the water and a powder was finally obtained. The powder was thoroughly dried in an oven at 55° C. overnight. The powder was then ground in a hammer mill.

### Example II

The procedure of Example I was followed except that 10 parts of Marasperse CB were used in place of 10 parts of Tamol NNO.

### Example III

0.3 part Celliton Blue G Extra, 1,4,5,8-tetraaminoanthraquinone,



foreign prototype No. 62, and 0.6 part of a 50% dispersion of paraphenylphenol and Tamol NNO were pasted with 2 parts Igepon T ( $C_{17}H_{33}CONHC_2H_4SO_3Na$ ). 50 parts of warm (120° F.) water were added and the mixture was stirred for two minutes. 250 parts of cold water were added and 10 parts of boiled off fabric of the polyester of terephthalic acid and ethylene glycol entered into the dye vessel. The temperature of the

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contents of the dye vessel was raised to the boil in 15–20 minutes, with continual manipulation of the goods. The fabric was dyed at the boil for one and one-half hours after which time it was rinsed in cold water and dried. The fabric was dyed a level blue shade.

## Example IV

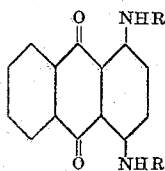
0.3 part of Celliton Fast Yellow 4RL Conc., benzene-azo-phenol,



was pasted with 2 parts Igepon T and 50 parts warm (120° F.) water were added. 0.6 part paraphenylphenol dispersed with 50% Tamol NNO was treated separately with 50 parts warm (120° F.) water and after stirring a few minutes, the dispersed dye solution was added. 200 parts of cold water were then added. 10 parts of boiled off fabric of polyester of terephthalic acid and ethylene glycol were entered into the dye bath and the temperature was raised to the boil. The fabric was dyed at the boil for one and one-half hours and then rinsed and dried. The fabric was dyed a level orange shade.

## Example V

The procedure of Example III was followed except that 0.3 part of Celliton Fast Blue FFRS,



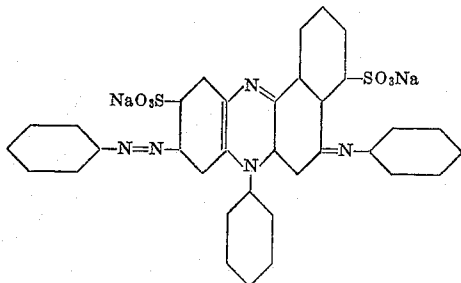
$R=CH_3$  and  $-C_2H_4OH$  mixture was used in place of 0.3 part Celliton Blue G Extra. A level dyeing resulted and the fabric was dyed a deep, bright blue shade.

## Example VI

The procedure of Example V was followed except that 0.6 part of paraphenylphenol dispersed with 50% Marsperse CB was used instead of 0.6 part of paraphenylphenol dispersed with 50% Tamol NNO. The fabric was dyed a bright blue shade and a level dyeing was obtained.

## Example VII

0.45 part paraphenylphenol dispersed with 50% Tamol NNO was stirred with 50 parts warm (120° F.) water and then 100 parts of cold water were added. 10 parts of fabric of vinyl chloride-acrylonitrile copolymer were manipulated in this bath for 5 minutes. To the bath were then added, successively with stirring, 0.5 part glacial acetic acid. 0.1 part cupric sulfate, 0.04 part zinc double salt of rongalite and 0.2 part Wool Fast Blue BLA Ex.-CF,



Color Index No. 228, all previously and separately dissolved in water. Enough water was added to make a total of about 300 parts and the temperature was raised to the boil for one and one-half hours with continued stirring. The dyed goods was rinsed in cold water and dried. A level dyeing resulted and the fabric was dyed a deep bright blue shade.

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## Example VIII

Ten parts of orthophenylphenol and ten parts of Tamol NNO were placed in a Werner Pfeleiderer type dispersion mill. Five parts water were added and the material was milled in a viscous state for two and one half hours. Cold water was run through the jacket of the mill to keep the temperature of the mix below the melting point (56° C.) of the orthophenylphenol. The milled material was dried in a vacuum oven at 50° C. The product was ground in a hammer mill.

## Example IX

The process in Example III was followed except that 0.6 part of a 50% dispersion of orthophenylphenol was used in place of 0.6 part of paraphenylphenol.

## Example X

The process in Example VII was followed except that 0.6 part of a 50% dispersion of orthophenylphenol was used in place of 0.6 part paraphenylphenol.

The dyeing process of the instant invention has many advantages over the dyeing processes of the prior art where dyeing assistants such as the phenylphenols are employed. Thus, it is much more convenient to the dyer to use dispersed powder of paraphenylphenol as it eliminates the necessity of first forming the sodium salt and then regenerating the phenol by addition of an acid. The possibilities of errors are minimized and the desired pH of the dyebath is more easily maintained. This is particularly important if acetate dyes are used. It is also a convenience and advantage to the dyer to add the dyestuff and the dispersed dyeing assistant simultaneously than to have to pretreat the textile structure to be dyed with paraphenylphenol as has been found necessary heretofore.

It is to be understood that the foregoing detailed description is given merely by way of illustration and that many variations may be made therein without departing from the spirit of our invention.

Having described our invention what we desire to secure by Letters Patent is:

1. In a process for dyeing a textile structure wherein the dyeing medium comprises a dyestuff for the material of the textile structure and a dyeing assistant composition, the improvement which comprises the use, in combination with the dyestuff, of a dyeing assistant composition consisting essentially of a dispersed powder prepared by milling a mixture of a phenylphenol selected from the group consisting of orthophenylphenol and paraphenylphenol, an anionic dispersing agent and water in a viscous state, passing steam through said mixture during the milling thereof to remove part of said water, continuing the milling of said mixture until a dry powder is obtained, subjecting said dry powder to the action of heat, and then grinding said powder to greater fineness.

2. In a process for dyeing a textile structure wherein the dyeing medium comprises a dyestuff for the material of the textile structure and a dyeing assistant composition, the improvement which comprises the use, in combination with the dyestuff, of a dyeing assistant composition consisting essentially of a dispersed powder prepared by milling a mixture of a phenylphenol selected from the group consisting of orthophenylphenol and paraphenylphenol, a sodium lignin sulfonate dispersing agent and water in a viscous state, passing steam through said mixture during the milling thereof to remove part of said water, continuing the milling of said mixture until a dry powder is obtained, subjecting said dry powder to the action of heat, and then grinding said powder to greater fineness.

3. In a process for dyeing a textile structure wherein the dyeing medium comprises a dyestuff for the material of the textile structure and a dyeing assistant composition, the improvement which comprises the use, in com-

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ination with the dyestuff, of a dyeing assistant composition consisting essentially of a dispersed powder prepared by milling a mixture of a phenylphenol selected from the group consisting of orthophenylphenol and para-phenylphenol, a sodium lignin sulfonate dispersing agent and water, in a viscous state, passing steam through said mixture during the milling thereof to remove part of said water, continuing the milling of said mixture, until a dry powder is obtained, subjecting said dry powder to the action of heat, and then grinding said powder to greater fineness.

4. In a process for dyeing a textile structure wherein the dyeing medium comprises a dyestuff for the material of the textile structure and a dyeing assistant composition, the improvement which comprises the use, in combination with the dyestuff, of a dyeing assistant composition consisting essentially of a dispersed powder prepared by milling a mixture of a phenylphenol selected from the group consisting of orthophenylphenol and para-

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phenylphenol, a sodium salt of a naphthalene sulfonic acid-formaldehyde condensation product dispersing agent and water in a viscous state, passing steam through said mixture during the milling thereof to remove part of said water, continuing the milling of said mixture until a dry powder is obtained, subjecting said dry powder to the action of heat, and then grinding said powder to greater fineness.

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