

UNITED STATES PATENT OFFICE

2,690,953

FUGITIVE TINTING OF NYLON FIBERS

John E. Livak and Lamont Hagan, Clemson, S. C.,
assignors to Deering Milliken Research Trust,
Pendleton, S. C., a nonprofit trust of Maine

No Drawing. Application December 28, 1950,
Serial No. 203,216

7 Claims. (Cl. 8—56)

1

This invention relates to the fugitive tinting or coloration of textiles, more particularly those based on wool, nylon and other synthetic fibers, including those which are acrylonitrile homopolymers, or acrylonitrile copolymers, or polyesters, e. g., polyesters from terephthalic acid and glycols, but excluding those which are cellulosic or cellulose derivatives. The invention is also applicable to textile yarns based on blends of wool with such non-cellulosic synthetic fibers.

Fugitive coloration of nylon, in particular, presents special problems since the tinted yarn, or fabric woven or knitted therefrom, is subjected to a heat-setting treatment, usually with steam. This heat-setting treatment tends to fix the tinting dye more or less permanently in the fibers so that the yarns remain stained or colored, and the tint cannot be removed readily in later finishing operations, e. g., the scouring bath. Special reducing or oxidizing treatments to remove the tint are accordingly required. The above, as well as other factors, affect the fugitivity of dyes or tints on nylon or other yarn subjected to heat-setting. Accordingly, it cannot be predicted with certainty which dyes will be suitable for the fugitive tinting of such fibers.

According to the present invention, we have found that phenolic phthaleins, e. g., phenolphthalein, thymol phthalein and cresolphthalein, which are highly colored in alkaline solutions and colorless in acid solutions, are especially effective for fugitively tinting, from alkaline aqueous media, non-cellulosic synthetic fibers such as, nylon, polyacrylonitrile (Orlon), terephthalic acid ethylene glycol polymer (Fiber V), and acrylonitrile copolymer fibers (Dynel and Chemstrand), and blends of wool with such non-cellulosic synthetic fibers. Not only is the tint completely removable by scouring procedures commonly employed in textile processing, but in addition, it is completely decolorized when exposed to dilute acid media at room temperature.

In our experiments, we have found that in the use of phenolic phthaleins as such, there is the drawback that premature decolorization of the tint may occur before the tint has served its purpose of an identification agent. Such premature decolorization occurs upon drying of the tinted material, or when the tinted material is heated to elevated temperatures, or permitted to stand in the air for long periods of time. We have discovered that when the alkaline earth metal salts, e. g., the calcium, strontium and particularly the barium salts of the phenolic phthaleins are employed instead of the sodium salts

2

for tinting, the alkaline earth metal salts remain colored much longer than the sodium salts. Furthermore, we have found that the application of the alkaline earth metal salts together with a humectant, such as glycerine, ethylene glycol, sorbitol, and the like, extends considerably the period of color retention. The alkaline earth metals salts of the phenolic phthaleins can be employed per se or they can be formed in situ in the tinting composition by reaction between the phenolic phthaleins and the alkaline earth metal hydroxides, e. g., barium hydroxide and strontium hydroxide. It is to be understood that the tinting solutions may also contain the usual wetting agents, thickeners, penetrants and the like.

The following examples will serve to illustrate the novel tinting solutions comprising our invention and their application.

Example 1

400 grams of barium hydroxide octahydrate are dissolved in 545 grams of water and 300 grams of phenolphthalein are added to the resulting solution with good stirring. Stirring is continued until a homogeneous solution of the thus formed barium salt of phenolphthalein is obtained. To this solution are then added 225 grams of glycerine and then 30 grams of Triton X-100 (an alkylaryl polyether wetting agent). The thus prepared stock solution is fully stable. It can be diluted with water to give any tint concentration desired. For fugitive tinting the stock solution is diluted with water so that the concentration of the barium salt of phenolphthalein is about 0.1 per cent to about 0.5 per cent.

Example 2

A tinting solution is prepared by mixing together 215 grams of thymolphthalein, 175 grams of barium hydroxide octahydrate, 150 grams of glycerine, 40 grams of Triton N-100 (a non-ionic wetting agent), and 580 grams of water in the same manner as described in Example 1. This tinting solution contains the barium salt of thymolphthalein which formed in situ. The solution can be applied as such or can be further diluted with water to the desired tinting concentration.

Example 3

A tinting solution is prepared by mixing together 175 grams of o-cresolphthalein, 175 grams of barium hydroxide octahydrate, 175 grams of glycerine, 45 grams of Igepal CTA Extra (polymerized ethylene oxide-alkylphenol condensa-

3

tion product) as a wetting agent, and 570 grams of water, in the same manner as described in Example 1.

Example 4

A tinting solution is prepared by mixing together the following ingredients in the same manner as described in Example 1: 160 grams of phenolphthalein, 150 grams of strontium hydroxide octahydrate, 200 grams of 1 per cent sodium carboxymethyl cellulose solution, 65 grams of Alrosol C (non-ionic fatty amide liquid) as a wetting agent, and 575 grams of water.

The tinting solutions of Examples 3 and 4 can be employed as such or further diluted with water to the desired tinting concentration. In Example 3, the barium salt of o-cresolphthalein was formed in situ. In Example 4, the strontium salt of phenolphthalein was formed in situ. The tinting compositions illustrated by the above four examples each have a pH of above 8, and impart to the textile materials to which they are applied the characteristic color in alkaline medium of the particular phenolic phthalein.

The tinting solutions described in the above examples can be applied in any suitable manner. Thus, for example, they can be sprayed on loose or staple fibers. In the case of non-cellulosic synthetic fiber wool blends, e. g., nylon-wool blends, the tinting solutions are applied dropwise onto a roving of the blends, conveniently at the pin drafter. The tinting solutions are applied to 100 per cent wool stock preferably by spraying. Yarns can be passed through a bath of the tinting solution or passed over a wick applicator. The textile materials are tinted with the characteristic color of the particular phenolic phthalein employed.

When applied to nylon, or other yarn subjected to heat-setting, the tint can readily be removed therefrom even if the tinted fiber has been spun into high twist yarn and subjected to a steaming operation in order to set the twist.

After the tinted fibers and yarns have been processed, the tint is removed therefrom by simply scouring with soap and water, or by treatment with a dilute acid medium at room temperature, such as dilute sulfuric, hydrochloric or acetic acid.

It will be understood that the usual lubricants and sizes may be included in the tinting solutions,

4

if desired. The pH of the solution can also be varied as long as it is maintained sufficiently alkaline to give the colored form of the phenolic phthaleins. This is usually above a pH of 8. Changes in the concentration and relative amounts of the components can be made without departing from the spirit of our invention.

The following is claimed:

1. A method for fugitively tinting nylon textile materials in which said materials are subjected to steaming after tinting, which comprises subjecting the textile materials to an aqueous alkaline solution containing an alkaline earth metal salt of a monohydric phthalein as the tinting agent, and a humectant non-reactive with said phthalein salts and said textile materials, and said humectant comprising a polyhydric alcoholic compound, the solution being at a pH of above 8, said tinting agent being adapted to be completely removed from said textile material by a conventional soap and water scour.

2. A method in accordance with claim 1, wherein the tinting agent is an alkaline earth metal salt of a monohydric phthalein of the group consisting of phenolphthalein, o-cresolphthalein and thymolphthalein.

3. A method in accordance with claim 1, in which a nylon wool mixture is tinted.

4. A method in accordance with claim 1, in which barium salt of phenolphthalein is the tinting agent and glycerine is the humectant.

5. A method in accordance with claim 1, in which the barium salt of thymolphthalein is the tinting agent.

6. A method in accordance with claim 1, in which the barium salt of o-cresolphthalein is the tinting agent.

7. A method in accordance with claim 1, in which the strontium salt of phenolphthalein is the tinting agent.

References Cited in the file of this patent

UNITED STATES PATENTS

Number	Name	Date
1,520,891	Spurway	Dec. 30, 1924
1,852,137	Whitehead	Apr. 5, 1932
2,623,834	Armitage	Dec. 30, 1952

OTHER REFERENCES

Rayon and Synthetic Textiles for September 1949 (article by Uhler), pages 95, 96.