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DYEING VINYL CHLORIDE FIBERS WITH
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1 Claim. (Cl. 8-55)

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This invention relates to a method of dyeing synthetic fibers after formation thereof. More particularly, the invention is concerned with the dyeing of polymerized vinyl ester fibers, including polymerized vinyl monochloride (commonly known as vinyl chloride) and polymerized vinyl dichloride (commonly known as vinylidene chloride), and both hereinafter generically referred to as "vinyl chlorides."

Vinyl chlorides are not readily water permeable and are, therefore, not susceptible to dyeing in any conventional manner. Heretofore, fibers of vinyl chlorides have been colored commercially by mixing dyestuffs with the raw materials fed into an extrusion machine and extruding a colored fiber. This method was not desirable, primarily because it did not afford a large enough selection of colors and could not be used to make small quantities of fibers of preselected colors to match fabrics, articles, etc.

Previous attempts to dye extruded fibers of vinyl chlorides have proven unsatisfactory, due to extreme weakening of the fibers or surface application only of the color.

It is an object of my invention to provide a method for dyeing fibers of polymerized vinyl chlorides after their formation, which method does not weaken the fibers greatly, if at all, will dye the fibers through and through and is extremely rapid and economical in operation.

Other objects of the invention will in part be obvious and in part hereinafter pointed out.

My method essentially consists in employing dyes which are characterized by their transformation to a leuco stage upon alkalization and boiling. In carrying out the method, one or more such dyes, selected to obtain a desired color, are dissolved in water, with the assistance of a solubilizing agent if necessary, to form a colored dye solution or bath. The bath is alkalized, after the dye is dissolved, and boiled to transform the dye to its leuco stage. This causes the bath to lose its previous color, and the same usually turns a clear water-white. If fibers of vinyl chlorides are immersed in such a hot leuco dye bath, the fibers will be dyed rapidly, even while the bath remains colorless, the intensity of the imparted color depending upon the length of immersion of the fibers, and the temperature and strength of the bath. If desired, an oxidizing agent may also be included to speed up the dyeing action of the bath.

The fibers of polymerized vinyl chlorides may be dyed, according to the method outlined above, either in their original fibrous state, e. g. skeins,

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when partially fabricated, e. g. as woven cloth or knitted fabric, or when fully fabricated, e. g. as a sweater or dress.

Colorless or tinted transparent extruded fibers of polymerized vinyl chlorides are dyed readily, pursuant to my invention. However, opaque extruded fibers of vinyl chlorides may also be dyed in the same manner, provided they are white, or, if a color, then lighter than the shade to be dyed.

The fibers may comprise either of the vinyl chlorides, i. e., vinyl monochloride or vinyl dichloride, or may be a copolymer of these two, or a polymer of either or both and some other substance, for example, vinyl acetate.

Dyes of the type aforementioned, to wit, those which are transformed to a leuco stage upon alkalization and boiling include:

Crystal violet, methyl violet, Fuchsine, malachite green, and auramine (known as the aryl methane dyes); methylene blue; chrysoidine; and substitution products and derivatives of the foregoing.

All of the foregoing specifically enumerated dyes, except chrysoidine, will, upon dissolving in alkali solution and boiling, form a bath which is substantially clear or water-white. A chrysoidine bath will, under the same circumstances, turn from orange to light amber. By "leuco" as used herein, therefore, I mean to include those dyes whose baths pale but do not completely decolorize.

Some of the dyes above mentioned do not dissolve as fast and as completely in water as is desired and it is, therefore, preferable to employ a solubilizing reagent to facilitate dissolving the same in water. To this end I may employ a water miscible organic solvent, such as alcohol, acetin, or acetic acid, which has the ability to dissolve a dye of the type under consideration and is itself miscible in water.

For the alkalizing reagent, I may employ any well known substance which is able to maintain the pH of the solution above seven and preferably between ten and eleven. Examples of suitable alkalizing reagents are potassium carbonate, sodium hydroxide and sodium oleate. It may be mentioned that when sodium hydroxide is employed, the dye bath in its leuco state has a brownish rather than a water-white cast.

For the oxidizing reagent, any well known oxidizing substance which is soluble in the dye bath may be employed. Examples thereof are potassium bichromate and sodium chlorate.

Dyeing is effected at any bath temperature. However, it proceeds most rapidly at higher

temperatures and I prefer to use bath temperatures ranging from 80 to 100° C. Highly satisfactory results are obtained at 80° C., at which temperature the fibers are not weakened at all if vinyl chlorides are dyed. With a boiling dye bath, the time of dyeing is very short but the fiber strength of vinyl chlorides is somewhat reduced.

By way of example and without in any manner limiting myself to this form of the invention, I have given below an example of one method which may be employed to dye fibers of vinyl chlorides:

Prepare two stock solutions, one consisting of a two per cent. solution of sodium chlorate (2 grams of sodium chlorate to 100 cc. of water) and the other a ten per cent. solution of sodium carbonate. A suitable dyestuff or combination of dyestuffs is then selected according to the color into which the fibers are to be dyed. A small amount of this dyestuff or mixture of dyestuffs, for example, one-half of a gram, is dissolved in alcohol, e. g. 50 cc. of denatured ethyl alcohol of normal strength (about 90 proof), or of methyl or isopropyl alcohol. To 5 cc. of the alcohol dye solution there is added 20 cc. more of alcohol (preferably the same kind of alcohol), in order to make sure that the dye is dissolved. To this mixture there is added 20 cc. of the sodium chlorate solution, 20 cc. of the sodium carbonate solution and 70 cc. of water. The final mixture is the dye bath and has the color of the dyestuff or combination of dyestuffs. Said dye bath will also be of the color it is desired to dye the fibers. The dye bath is brought to a boil and kept boiling until the color of the dye bath completely changes, pales or disappears. When the dye bath is composed of the foregoing substances, in approximately the specified proportions, the color of the solution after a suitable period of boiling (three to five minutes) will be water-white, i. e. absolutely clear or very pale. This is the leuco state of the bath and results from transforming the dyestuff used to a leuco stage or leuco base. The bath is now ready for dyeing, which is effected by immersing the fibers of vinyl chlorides into the leuco state dye bath while maintaining the bath hot. As the fibers are watched, it will be seen that they take on the desired color, even as the bath remains in its leuco state. The intensity to which the fibers are dyed will depend upon the length of the immersion in the hot leuco dye bath and upon the temperature and strength of both. I have found that about a four minute immersion period is satisfactory to create a good depth of color at boiling and one and one-quarter hours for the same depth of color at an 80° C. bath temperature. Of course, a lesser or more extended immersion may be necessary, depending upon the desired intensity, and the bath temperature employed is preferably the highest which does not appreciably affect the fibers of vinyl chlorides being dyed. The bath may be used until the dye is exhausted therefrom. Since the bath must be kept hot to effect rapid dyeing, the fibers may be pulled out of the bath at any time to observe and halt the progress of the dyeing. Dyeing will be resumed when the fibers are again submerged in the hot bath.

Fibers of vinyl chlorides dyed in accordance with the foregoing method will be found to be

dyed throughout and the fibers not appreciably weakened or distorted during or by the dye operation. However, there is sometimes a tendency for the dyed fibers to crock, fade and bleach. It is, therefore, desirable to set the dye. I have found that setting may be satisfactorily effected by heating the fibers to a temperature of from about 80 to 110° C. and maintaining the same at said temperature for about one minute. Such heat setting may be satisfactorily performed in a heated oil bath. For this purpose, I employ any oil having a relative high flash point, i. e. sufficiently above 110° C., so that there will be no danger of fire or explosion. Examples thereof are castor oil, machine oil and cutting oil. Such an oil is placed in a fixing tank and raised to a temperature of about 70° F. The dyed fibers after having water from the dye bath shaken off, drying being unnecessary, are immersed in the oil which is then heated at any speed to from about 80 to 110° C. but no higher. The fibers are allowed to remain about one minute in the heated bath, or longer if desired, and are then taken out and the oil cleaned off. This may be done either by emulsification, i. e. with soap and water, or by dissolving the oil in a suitable solvent which does not affect fibers of vinyl chlorides, carbon tetrachloride being an example thereof.

It will thus be seen that I have provided a method of dyeing fibers of vinyl chlorides so as to achieve the several objects of my invention.

As various possible embodiments might be made of the above invention, and as various changes might be made in the embodiment above set forth, it is to be understood that all matter herein set forth is to be interpreted as illustrative and not in a limiting sense.

Having thus described my invention, I claim as new and desire to secure by Letters Patent:

A method of dyeing fibers essentially comprising polymerized vinyl chlorides, said method comprising dissolving in water an alkalizing reagent constituting an alkali metal salt and a basic dye capable of being transformed to a leuco stage upon alkalization and boiling, boiling the water with the dissolved dye to form a leuco dye bath, immersing the fibers in said bath to dye the same while the bath remains in its leuco state, and then removing said fibers from the bath.

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