United States Patent Office 3,211,755 Patented Oct. 12, 1965

3,211,755

1-AMINO-2-BENZOYL-4-HYDROXY ANTHRAQUINONE

Joseph William Fitzpatrick, Pine Beach, Philip Wehner, Toms River, and John David Hildreth, Beachwood, N.J., assignors to Toms River Chemical Corporation, Toms River, N.J., a corporation of Delaware

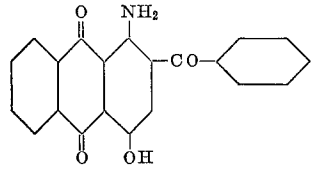
No Drawing. Filed Sept. 27, 1963, Ser. No. 311,986
1 Claim. (Cl. 260—376)

This invention relates to a novel blue disperse dyestuff of the anthraquinone series and to the use of this dyestuff in coloring polyester materials. More particularly, this invention relates to 1-amino-2-benzoyl-4-hydroxy anthraquinone and to its use in coloring polyester fibers.

Polyester fibers present particular dyeing problems, arising at least in part out of the hydrophobic nature of such fibers. In the dyeing of polyester fibers, the class of dyes known as disperse dyestuffs has come to have the widest application. These dyestuffs are essentially water-insoluble products applied in a finely divided condition from a dispersion. In the application of such dyestuffs, the dyeing difficulties associated with polyester fibers have been met by the development of special methods for the application of disperse dyes to the fibers. Of these methods, the one known as the Pad/Thermofix method has become of increasing importance since it is particularly adapted for high-speed, continuous dyeing operations. In this method, a fabric is padded by passing it through an aqueous suspension of the dyestuff and squeezing the fabric between closely-set rollers in order to remove excess dye liquor. The dyestuff is only loosely attached to the fiber at this point. The dyestuff is then fixed on the fiber by subjecting the material to a short, intensive heat-treatment at elevated temperatures of the order of about 120–220° C. It is evident that a dyestuff, in order to be suitable for application by this method, must be fast to sublimation or else it will wholly or partially volatilize from the fiber during the heat-treatment step. The result of such sublimation will be a loss of color value on the polyester fiber and, if a union dyeing operation is being carried out, the staining of the other fibers, such as cotton, which may be present in the blend.

The problems associated with the dyeing of polyester fibers, especially polyethylene terephthalate, have been particularly troublesome with respect to the provision of suitable blue dyestuffs for this purpose with satisfactory fastness to light.

In accordance with the present invention, it has been found that 1-amino-2-benzoyl-4-hydroxy anthraquinone of the formula:



gives blue dyeings on polyester fibers of excellent shades and of excellent fastness to light and to sublimation. The dyestuff of this invention is very readily applicable by the Pad/Thermofix method and provides good tinctorial value and strength build-up.

The dyestuff of this invention is of particular interest in that it provides blue dyeings of excellent shade and light fastness on polyester fibers such as polyethylene terephthalate. Blue dyestuffs of satisfactory light fastness properties for use in coloring polyester fibers are difficult to obtain.

For dyeing, the said dyestuffs are preferably used in a finely divided form and the dyeing is carried out in the

presence of a dispersing agent such as sulfite cellulose waste liquor or a synthetic detergent, or a combination of different wetting and dispersing agents. Before dyeing, it is generally of advantage to convert the dyestuff or dyestuffs to be used into a dyestuff preparation which contains a dispersing agent and the finely divided dyestuff(s) in such a form as to yield a fine dispersion when the preparation is diluted with water. Dyestuff preparations of this kind can be made by known methods, for example, by grinding the dyestuff(s) either in dry or wet form with or without the addition of a dispersing agent.

The dyestuff of the present invention is particularly adapted for dyeing by the so-called thermofixation or Pad/Thermofix method, in which the fabric to be dyed is impregnated advantageously at a temperature not exceeding 60° C. with an aqueous dispersion of the dyestuff, which may contain 1 to 50% of urea and a thickening agent, especially sodium alginate, and the fabric is squeezed in the usual manner. The squeezing is preferably carried out so that the goods retain 50 to 100% of their weight of dye liquor.

The dyestuff is fixed by subjecting the impregnated fabric to a heat treatment at temperatures above 100° C., for example, at a temperature ranging from 120–220° C., it being of advantage to dry the fabric prior to this treatment, for example, in a current of warm air.

The thermofixation mentioned above is of special interest for the dyeing of mixed fabrics of polyester fibers and cellulose fibers, especially cotton. In this case, in addition to the dyestuff to be used in the process of the invention, the padding liquor contains a dyestuff suitable for dyeing cotton, for example, a direct dyestuff or vat dyestuff, or more especially a so-called reactive dyestuff, i.e. a dyestuff capable of being fixed on cellulose fibers with the formation of a chemical bond, for example, a dyestuff containing a chlorotriazine or chlorodiazine residue. In the latter case it is of advantage to add to the padding liquor an agent capable of binding acid, for example, an alkali carbonate, alkali phosphate, alkali borate or alkali perborate, or a mixture of two or more of these agents. When vat dyestuffs are used the padded fabric must be treated, after the heat treatment, with an aqueous alkaline solution of a reducing agent of the kind used in vat dyeing.

The dyeings produced on polyester fibers by the process of the invention are advantageously given an after-treatment, for example, by heating them with an aqueous solution of a non-ionic detergent.

Instead of applying the dyestuffs in the process by impregnation, they may be applied by printing. For this purpose, a printing colour is used which in addition to the usual printing assistants such as wetting and thickening agents, contains the finely dispersed dyestuff, if desired, in admixture with one of the foresaid cotton dyestuffs, and, if desired, in the presence of urea and/or an agent capable of binding acid.

There are obtained by the practice of the present invention strong dyeing or prints having excellent fastness properties, especially a good fastness to sublimation and to light.

The term polyester defines synthetic polymeric polyesters such as the highly polymeric linear polyesters, the molecules of which have recurring monomeric units connected by ester linkages. Dibasic acids, for example, aromatic acids such as terephthalic acid, diphenyl-4,4'-dicarboxylic acid and/or diphenylsulfone-4,4'-dicarboxylic acid and dihydroxy compounds, for example, glycols such as ethylene glycol, diethylene glycol, triethylene glycol, propylene glycol and/or butylene glycol, as well as other diols, such as 1,4-cyclohexyldiol can be used as the monomers to form the polymeric polyesters. Typical commercial examples of such fibers are Dacron, Terylene, Fortrel, Trevira, Terlanca, Kodel, Vycron, etc. They are

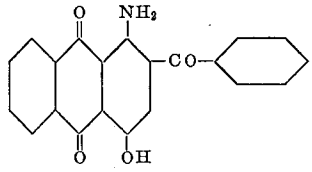
disclosed, for example, in U.S. Patent No. 2,901,466 and British Patents Nos. 578,079, 579,462, 588,411, 588,497 and 596,688.

The present invention is, of course, equally applicable to the dyeing of blends of polyester fibers and cellulosic fibers. The latter term includes native cellulose, such as linen or more particularly cotton, as well as regenerated cellulose, such as viscose or cuprammonium rayon.

The following examples illustrate the invention, the parts and percentages being by weight.

Example 1

10.7 parts of boric acid are dissolved in 320 parts of 5% oleum at 50–60° C. with stirring. 30 parts of 1-amino-2-benzoyl-4-bromo anthraquinone are added slowly and the reaction mixture heated slowly to 100° C. over 1 hour and held at 100° C. for a further period of 1 hour. The reaction mixture is then heated quickly to 120° C. and held at 120° C. for 5 hours, dry air being blown through the reaction mixture to remove the bromine gas liberated. At the end of this period, the reaction mixture is cooled to 60° C. and then drowned in 1,000 parts of cold water. The resulting mixture is suction-filtered at 60° C. and then washed with cold water until neutral, followed by drying with warm air. The product, 1-amino-2-benzoyl-4-hydroxy anthraquinone, is obtained as a blue powder in a yield of 26 parts and has the formula:



Example 2

10 parts of the dyestuff of Example 1 are brought to a state of fine dispersion by milling in a ball mill with 2.5

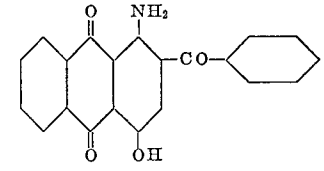
parts of the condensation product of naphthalene-2-sulfonic acid with formaldehyde, 7.5 parts of sorbitol (other polyalcohols can also be employed), and 50 parts of water.

An amount of this preparation sufficient to provide a concentration of 3 grams of dyestuff per liter of dye bath is finely dispersed in water containing 0.5 g. of sodium alginate per liter and the resultant pad liquor is brought to a temperature of 25° C.

A polyester fabric from ethylene glycol and terephthalic acid is then padded with the above liquor and mechanically squeezed to a 65% pick up. The padded material is then air dried and developed by dry heat curing for 1 minute at 200° C. The dyed fabric is then cold rinsed, scoured and finally dried. A bright blue dyeing of excellent light and sublimation fastness and good penetration characteristics is obtained.

What is claimed is:

1-amino-2-benzoyl-4-hydroxy anthraquinone of the formula:



References Cited by the Examiner

UNITED STATES PATENTS

2,773,071	12/56	Pizzarello	260—380
2,967,752	1/61	Bucheler	260—376 X

FOREIGN PATENTS

661,045	11/51	Great Britain.	

LORRAINE A. WEINBERGER, *Primary Examiner.*

LEON ZITVER, *Examiner.*