United States Patent Office

3,100,133

Patented Aug. 6, 1963



3,100,133

# METHOD FOR DYEING POLYPROPYLENE TEXTILE MATERIAL AND PRODUCT

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No Drawing. Filed Dec. 30, 1960, Ser. No. 92,728
Claims priority, application Germany Jan. 12, 1960
5 Claims. (Cl. 8—55)

This invention relates to a method of dyeing textile materials composed of polypropylene and to the resulting dyed product which is characterized by its red color and its fastness to light and rubbing.

The textile art over a number of years has seen the introduction of a large number of artificial and synthetic fiber-forming polymers to complement and often to replace natural fibers such as wool, cotton and silk. Viscose rayon and cellulose acetate are typical examples of artificial substances employed for this purpose, and well known examples of synthetic fibers are the polyamides, polyesters, polyacrylonitrile and the like. All of these substances have not only the requisite properties for forming fibers but are also capable of being dyed in a relatively simple manner. Thus, many thousands of dyestuffs have been discovered and used by the textile industry to obtain dyed fabrics and the like of almost any desirable color with variations in shade, brilliance or similar properties. Over a period of time, the fastness properties of these dyestuffs have also been improved, although fastness depends not only on the dyestuff itself but very often also depends on the particular material being dyed.

In more recent years, there has been considerable interest in the utility of polypropylene as a synthetic textile polymer, because of its relatively high molecular weight and highly oriented linear chain. In spite of the valuable physical properties of polypropylene polymers when stretched or drawn in suitable textile form, it is very difficult and almost impossible to impart a color to the textile product by using the more usual dyebath techniques of this art. The reason for this is that a polypropylene textile material has an especially poor affinity for dyestuffs, e.g. when compared to most of the other known synthetic materials discussed above.

Therefore, the art has generally employed the so-called spin dyeing or internal dyeing process whereby a finely divided coloring agent, usually a pigment, is dispersed in the liquid polypropylene polymer before it is spun or drawn into textile form. This process has many limitations and disadvantages for both the producer of the raw filament and also the textile dyer and manufacturer as well as the consumer, because internal dyeing lacks flexibility in adjusting the supply and demand for certain colors and prohibits independent control of the goods by the dyer. Furthermore, once the pigment is locked into the polymer by internal dyeing, it cannot be easily removed by bleaching or stripping operations.

There are actually only a very few dyestuffs which are known to be capable of dyeing a polypropylene textile material from a dyebath. For example, there is a limited number of compounds in the form of azo dyestuffs containing long chain alkyl substituents which yield yellow and green dyeings when applied to polypropylene. Otherwise, there appears to have been very little success in the discovery of dyestuffs for use in combination with polypropylene.

A principal object of the present invention is to provide a new and useful method for dyeing a polypropylene textile material so as to obtain a red colored product with very good fastness properties.



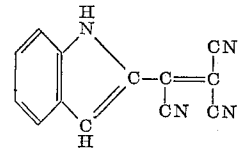
Another object of the invention is to provide a method for dyeing a polypropylene textile material by a simple dyebath technique which is relatively easy to regulate and more convenient for industrial dyers.

Yet another object of this invention is to provide a new and useful composition in the form of a red dyed polypropylene textile material, thereby extending the now limited utility of this particular polymer.

Other objects and advantages of the invention will become more apparent upon a consideration of the following specification and description.

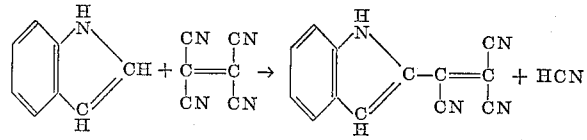
The term "textile material" is employed herein to designate all polypropylene structures which are common in the textile art such as: fibers and filaments, whether in the form of monofilaments, staple filaments or collected bundles as in yarns, tows, threads, ropes and the like; films; ribbons; tapes; felts; fabrics whether woven or unwoven; or finished products containing said polypropylene such as articles of clothing, blankets and the like. The term "dyestuff" is employed to designate a compound capable of imparting a color when applied to a textile material or structure. By comparison, a "pigment" is generally considered to be a finely divided insoluble material most easily incorporated prior to forming the textile structure from a liquid polymer. The term "elevated temperature" is employed herein with reference to temperatures substantially above room temperature.

In accordance with the present invention, it has now been discovered that a red colored polypropylene textile material with very good fastness to light and rubbing can be obtained by contacting the textile material at elevated temperature with an inert organic solvent containing as the effective dyestuff the compound of the formula



and then applying steam to the material at an elevated temperature, preferably saturated steam at a temperature of about 110° C. to 140° C., for a period of time sufficient to fix or develop the dyestuff compound on the polypropylene textile, e.g. about 20 minutes to 2 hours, preferably about 30 minutes to one hour.

The dyestuff compound is conveniently prepared by reacting indole with tetracyanoethylene in an inert organic solvent at elevated temperature, preferably between about 70° C. and 100° C., according to the following reaction scheme:



As indicated by the reaction scheme, the tetracyanoethylene and indole are preferably reacted in about equal parts by weight, i.e. in approximately molar equivalent amounts, although it is also possible to employ an excess of one of the reactants such as a tenfold to twentyfold excess by weight of tetracyanoethylene. The tetracyanoethylene is first dissolved in a suitable inert solvent such as benzene. The indole is then added and the reaction mixture is heated to a temperature between about 70° C. and 100° C., the reactants combining quite readily to form the dyestuff in solution. For example, one part by weight of tetracyanoethylene can be reacted with one part by

weight of indole in about 150 parts by weight of benzene while heating at a temperature of 80° C.

The resulting dyestuff can be readily separated from the solvent, but it is a particularly advantageous feature of the invention that the dyeing of the polypropylene textile material can be directly carried out in the reaction product solution without first recovering the dyestuff. Also, no other materials need be added to the inert solvent containing the dyestuff compound and no special pretreatment of the polypropylene material is required. The same inert organic solvents employed in preparing the dyestuff are equally useful as dyebath solvents. Accordingly the invention provides a very convenient method of dyeing because the dyestuff can be prepared in situ in the dyebath, merely by adding the two reactants and heating for a very short period of time. As the dyebath becomes exhausted of dyestuff during dyeing, the bath can thus be quickly and economically regenerated by the direct preparation of additional quantities of dyestuff therein.

The preferred solvents of the invention are accordingly those which will provide an inert reaction medium for preparation of the dyestuff, i.e. a solvent for tetracyanoethylene which does not otherwise substantially inhibit or enter into the reaction with indole. The tetracyanoethylene apparently forms an unstable complex with such solvents as benzene, but this complex does not affect a quantitative reaction with the indole. Because of the essentially inert property of the solvent, those skilled in the art can readily select suitable materials. In general, hydrocarbon solvents are preferred such as aromatic and partially hydrogenated aromatic hydrocarbons which may contain lower alkyl substituents, e.g. benzene, toluene, xylene, decalin and tetralin. Benzene is particularly useful because of an appropriate boiling point and its relatively low cost and availability. As noted above, these solvents are equally useful as a reaction medium in preparing the dyestuff and as a dyebath medium for applying the reaction product to the polypropylene textile material.

When using benzene, it is possible to work at atmospheric pressure and maintain the solvent at about its boiling point of 80° C. With solvents having a higher or lower boiling point than benzene, the reaction to produce the dyestuff and the subsequent dyeing can be carried out in a vacuum or at elevated pressure for adjustment of the boiling point. In this manner, the preferred temperature conditions can be accurately maintained for uniform results and a relatively large class of suitable solvents can be employed. Such variations in preparing the dyestuff and dyeing the textile are not critical but merely represent routine techniques in adapting particular solvents to a preferred operation.

The dyestuff can first be prepared in a relatively concentrated form with respect to the solvent, e.g. about 50 to 100 parts by weight of solvent to one part by weight of the dyestuff. When dyeing, this reaction product mixture is preferably diluted, although the concentration of the dyestuff in the dyebath can be varied over a wide range. Very good results have been obtained with a dyestuff concentration of about 0.001 to 0.005% with respect to the total weight of the dyebath, e.g. about 1 to 5 grams of dyestuff for every kilogram of solvent. The weight ratio of the polypropylene textile goods being dyed to the dyebath liquor is preferably in a range of about 1:30 to 1:50.

The textile goods are preferably immersed in the dyebath at a temperature between about 70° C. and 100° C. and for a period of time of about 15 to 60 minutes. Again, these conditions are subject to variation depending upon the desired color effect. Excess solvent is then removed from the textile material impregnated with the dyestuff compound, and the material is then subjected to a steam treatment in order to fix the dyestuff to the polypropylene. This fixing treatment is preferably carried out with saturated steam at the above-mentioned temperatures, preferably in the neighborhood of about 130° C. In general the steam treatment requires about 30 minutes,



but longer periods of treatment are not harmful and a total period of 2 hours should be sufficient in practically all cases. The dyed and fixed textile is finally washed with soap and acetone in the usual manner to remove the remaining solvent, and other finishing treatments may be employed as are common in the textile art.

The invention is further illustrated but is not limited by the following example which discloses one preferred method of obtaining desirable results.

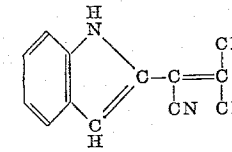
*Example*

10 grams of tetracyanoethylene is dissolved in 1 liter of benzene. There is then added about 1 gram of indole thereto and the solution is heated to about 80° C. A fabric of polypropylene fibers is dyed in this solution for about 15 minutes while maintaining the temperature at 80° C. The fabric is then withdrawn from the bath, excess solvent being removed, and subjected for about 30 minutes to a continuing treatment with saturated steam at 125° C. The textile material exhibits an intense brick-red dyeing. After washing with soap and acetone, fastness tests are carried out in accordance with the German "DIN"-standards as set forth in "Tests for Colour Fastness of Textiles, Part 11 (Light-fastness) and Part 18 (Rubbing-fastness), draft ISO, No. 117." The light fastness has a value of 6, the rubbing fastness (dry) has a value of 4 and the rubbing fastness (wet) also has a value of 4.

As fully illustrated by the foregoing example, the present invention provides a specific but excellent method for dyeing polypropylene textiles in a fast red color. Because of the very limited number of dyestuffs previously found for polypropylene, this invention represents an important step in advancing the art and providing a wider and more acceptable use of polypropylene fibers, fabrics and the like.

The invention is hereby claimed as follows:

1. A method for dyeing a polypropylene textile material which comprises contacting said material at elevated temperature with an inert organic solvent containing the compound of the formula

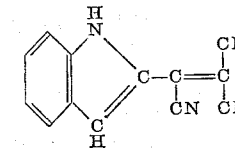


and then applying steam to said material to fix said compound as a dyestuff thereon.

2. A method as claimed in claim 1 wherein the inert organic solvent is benzene.

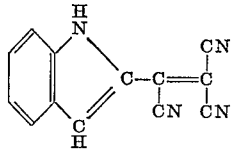
3. A method as claimed in claim 1 wherein the polypropylene textile material is immersed in said solvent containing said compound for a period of about 15 to 60 minutes while heating the solvent to maintain its temperature between about 70° C. and 100° C.

4. A dyed polypropylene textile material to which there has been applied and fixed by steam the compound of the formula



thereby providing a red colored textile material which is fast to light and rubbing.

5. A method for dyeing a polypropylene textile material which comprises contacting said material at a temperature of about 80° C. with benzene containing the compound of the formula





and then applying steam to said material at a temperature of about 110° C. to 140° C. to fix said compound as a dyestuff thereon.

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