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SPUNDYED RAYON

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This invention relates to the manufacture of artificial products dyed with vat dyes, such as threads, fibers, ribbons, films and the like, which are obtained by coagulation and decomposition of an alkaline spinning solution such as viscose solution.

The invention more particularly relates to a process for manufacturing artificial products, wherein a dye stuff derived, for example, from an indanthrene dye is dissolved in an alkaline spinning solution such as viscose. The solution thus formed is then spun to products, whereby during or after the spinning process, the dye stuff is oxidized in the products to develop the color.

It has heretofore been proposed to reduce the vat dye in an alkaline medium, for example, in caustic soda solution, by means of a reducing agent, for example, sodium hydrosulphite. The mixture obtained is mixed as such with a spinning solution, after which the solution is processed to products. In the practice of this process, besides the leuco compound of the vat dye which is soluble in alkali, various salts are formed in the spinning solution which exert an undesirable influence on the manufacture of the products and on the products themselves. According to the present invention, this undesirable influence can be prevented in a relatively simple way.

It has also been proposed, in Lockhart Patent No. 1,865,701, to reduce the vat dye by dissolving an alkaline paste of the same in viscose solution. However, it was found that indanthrene dyes are, in general, not reduced by the reagents present in viscose solution and patentee's process is for all practical purposes incapable of being operated on an appreciable scale with these dyes.

This invention has as an object to provide a process for manufacturing artificial products dyed with vat dyes in which the vat dye is introduced into a spinning solution in a stable form. A further object is to provide a process for manufacturing artificial products dyed with vat dyes during which no clogging of the spinneret orifices occurs. A still further object of this invention is to provide a process for manufacturing viscose rayon in which the color of the rayon produced is perfectly even. Other objects will appear hereinafter.

These objects are accomplished in the present invention by dissolving a purified free leuco compound of a vat dye in the alkali spinning solution, and thereafter processing the solution to products in the normal way.

In order to obtain the purified free leuco compound, the vat dye stuff is first reduced. The reduced dye solution is then acidified until it just shows an acid reaction. Formic acid or acetic acid have been found to be very suitable for this purpose. The acid should be added slowly in order to obtain a good precipitate which settles and can be easily separated. For example, it has been found that a good precipitate is obtained when the acid is added over a period of time varying from twenty minutes to a few hours, depending on the quantity of dye stuff being processed, the amount of stirring, etc. By regulating the acid addition and the degree of stirring, particularly in the critical range when the color changes,

the best results are achieved with regard to the quality of the precipitate.

The precipitate, consisting of the free leuco compound, can be purified in various ways, for example, by dialysis. More simply, however, it may be purified by carefully washing with water. If desired, the oxidizing action of the atmosphere may be eliminated by adding a small quantity of a reducing agent to the wash water.

The purified free leuco compound thus obtained is then mixed with water and dissolved in viscose solution while excluding atmospheric oxygen. If desired, this operation may be conducted under oxygen-free nitrogen gas. The addition of the purified free leuco compound mixed with water may be accomplished with particularly good results by injecting it into the viscose spinning solution a short while before spinning. For example, the leuco compound paste may be injected as the viscose passes through the pipe line near the spinning machine. The leuco compound dissolves very rapidly in the viscose and successful results have been obtained by injecting the paste into the viscose only a few feet from the spinning pump.

If there should be in the original vat dye a wetting agent or a dispersing agent, it must be removed or inactivated prior to the precipitation of the free leuco compound. For example, if there should be present an anion-active compound as a dispersing agent, a cation-active substance may be added to inactivate the dispersing agent.

Any desired quantity of the free leuco compound may be injected into the spinning solution. In general, it has been found that the present process requires less dye stuff than is required in pigment dyeing to obtain an equivalent effect. Lustrous products are obtained in the practice of the present invention and if less lustre is desired, a delustering agent may be added to the viscose along with the purified free leuco compound.

The following examples will further illustrate how the present invention may be carried out in practice, but the invention is not limited to these examples. All percentages given are percentage-by-weight.

Example 1

Dyestuff:

Indanthrene Golden-Yellow G. K.—powder (I. G.) Schultz, Farbstofftabellen, No. 1286 C.

The dyestuff was reduced in the normal way as follows: 14 grams of the dyestuff was first stirred with a small quantity of water at 60° C., and thereafter the paste was diluted with hot water to a volume of 1400 cc. To this mixture was added 42 cc. of sodium hydroxide solution (38° Bé.) and 14 grams of sodium hydrosulphite. After 15 minutes the dyestuff was reduced. The free leuco compound was next precipitated by slowly adding 50 cc. of a 34% solution of formic acid in water, drop by drop, over a period of 30 minutes. A good precipitate of the free leuco compound, which had an orange-yellow color, was obtained. By decanting and then adding water to which a small amount of sodium hydrosulphite had been added, stirring, and repeating these operations twice, a technically pure paste was obtained.

The paste was diluted up to 200 cc. with water and dissolved in viscose solution in an atmosphere of nitrogen in such a quantity that the amount of dyestuff was 2%, calculated on cellulose. A Bordeaux-red colored viscose solution was obtained and this was deaerated for 4 hours and then spun. Upon spinning, a thread was obtained which became golden-yellow in color upon coming into contact with the air. The spinning was carried out according to the normal method for spinning viscose rayon, and aftertreatment of the spun product was applied according to the usual processes. The oxidative bleaching

agents effected a completion of the oxidation. The golden-yellow, high-lustrous yarn so obtained was dried in the normal way.

Example 2

Dyestuff:

Indanthrene Brown B. R. powder (I. G.) Schultz, Farbstofftabellen II, p. 127.

14 grams of the dyestuff was stirred with a small volume of hot water and the paste was diluted to 1400 cc. with water at 55° C. Next 140 cc. of 19.4% NaOH solution and 21 grams of sodium hydrosulphite were added in that order. After 15 minutes, during which time the temperature of the mixture was maintained at about 50° C., the dyestuff was reduced. The solution was then acidified by slowly adding 200 cc. of 17% aqueous formic acid solution. The precipitate which formed was filtered and washed with water containing a small amount of sodium hydrosulphite.

A paste was prepared by adding 200 cc. of water to the precipitate. This paste was injected in the same manner as in Example 1 into viscose solution. The spinning of the viscose rayon yarn and the further finishing was carried out in the normal way. The yarn obtained had a completely even brown color.

Example 3

Dyestuff:

Idanthrene Brilliant Scarlet R. K. powder (I. G.) Schultz, Farbstofftabellen, Erganzungsband II, p. 200

10 grams of the dyestuff was reduced in the same manner as in Example 1, using the following quantities of materials: 500 cc. of water, 25 cc. of 19.4% NaOH solution, and 5 grams of sodium hydrosulphite.

Acidification was carried out by slowly adding 50 cc. of 12% aqueous formic acid solution. Further treatments were the same as in Example 2, and a very evenly colored viscose rayon yarn was obtained.

Example 4

Dyestuff:

Indanthrene Red Brown G. R. powder (I. G.) Schultz, Farbstofftabellen, Erganzungsband II, p. 204.

The reduction of 10 grams of the dyestuff was carried out as in Example 2, using the following quantities of materials: 100 cc. of water; 50 cc. of 19.4% NaOH solution; and 10 grams of sodium hydrosulphite. The acidification was carried out by slowly adding 100 cc. of 17% formic acid solution. After washing the filtered precipitate twice, a paste was prepared and diluted to 200 cc. with water. This paste was injected into viscose solution used for spinning yarn. As in the other examples, very favorable results were achieved.

Example 5

Dyestuff:

Caledon Jade Green X. N. 300% powder (I. C. I.) Schultz, Farbstofftabellen, No. 1270.

This commercial product was found to contain a dispersing agent so that it was impossible to obtain a good precipitate of the free leuco compound by reducing and acidifying. To inactivate the dispersing agent, 20 grams of the dyestuff was stirred with 600 cc. of water. Then

150 cc. of a 2% lauryl pyridinium chloride solution was added and the mixture was heated to boiling. The mixture was allowed to stand for 4 hours, after which the dyestuff was filtered and washed.

The dyestuff was then reduced by adding the following materials in the order named: 600 cc. of water; 66 cc. of 19.4% NaOH; and 16 grams of sodium hydrosulphite. After 15 minutes at 50° C. the dyestuff was reduced after which it was acidified by slowly adding 50 cc. of 30% formic acid solution. The free leuco compound settled excellently and was technically pure after being washed twice by decantation and stirring. A paste made from the precipitate gave a clear dark green solution when dissolved in viscose. The colored viscose was spun to rayon yarn in the usual way. The yarn leaving the spinning bath was red and this color changed to green upon contact with the air during further processing of the yarn.

It will be apparent to those skilled in the art that this invention is not limited to the manufacture of viscose rayon yarn, but is applicable generally to the manufacture of artificial products dyed with indanthrene dyes which are obtained from an alkaline spinning solution.

The advantages of the present invention, both with regard to the spinning process and the products produced thereby, are numerous. The pure free leuco compound is far stabler than the reduced sodium salt in solution. When working with reduced solutions, the slightest trace of oxygen may give rise to the formation of undissolved oxidized particles, which may cause disturbances in the spinning processes or an uneven dyeing. With purified free leuco compounds, traces of oxygen do not immediately give rise to undesired oxidation, although these compounds are preferably stored while excluding oxygen. Since the purified free leuco compounds dissolve completely in the alkaline spinning solution, no clogging of spinneret orifices occurs. The color of the product manufactured according to the present invention is perfectly even.

What is claimed is:

1. A process of manufacturing viscose rayon yarn dyed with a vat dye comprising the steps of acidifying a reduced solution of a vat dye, separating the precipitated free leuco hydroxy compound, dissolving said purified free leuco compound in viscose spinning solution, spinning the resultant solution to form viscose rayon yarn, and oxidatively developing the dyestuff in the yarn.

2. The process of claim 1 wherein the purified free leuco compound is added to the viscose solution in the form of a paste.

3. A process of manufacturing viscose rayon yarn dyed with an indanthrene dye comprising the steps of acidifying a reduced solution of said dye, separating the precipitated free leuco hydroxy compound, adding a paste of said free leuco hydroxy compound to viscose spinning solution, spinning the resultant solution to form viscose rayon yarn, and oxidatively developing the dye stuff in the yarn.

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