Patented Jan. 31, 1939

2,145,580

UNITED STATES PATENT OFFICE

2,145,580

SPINNING SOLUTIONS CONTAINING ORGANIC DYES AND PRODUCTS THEREOF

Rudolph S. Bley, Milligan College, Tenn., assignor to North American Rayon Corporation, New York, N. Y., a corporation of Delaware

No Drawing. Application October 13, 1934, Serial No. 748,259. Renewed June 7, 1938

3 Claims. (Cl. 106—40)

The present invention relates to a process of producing dyed rayon products, such as filaments, yarns, ribbons, films, etc., by admixing suitable organic dyes with the spinning solutions prior to the extrusion thereof.

The primary object of my invention has to do with the addition of such organic dyes to strongly alkaline spinning solutions, such as viscose, cuprammonium cellulose, silk fibroin, alkali metal alginates, gelatine, etc., which will not be deleteriously affected by alkali or acids, desulphurizing agents and bleaching compounds used in the manufacture of artificial products from said spinning solutions.

Another object of this invention has to do with the incorporation of "Triazol-Echtrot C (Griesheim)", "Toluylenrot (Griesheim)", "Triazol-Reinblau R", etc., into strongly alkaline spinning solutions.

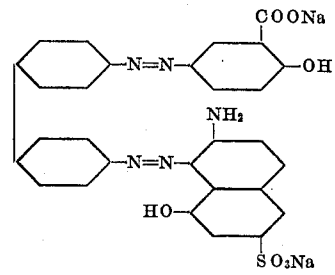
Other objects of my invention will become apparent to those skilled in the art after a study of the following specification.

I am well aware that it has, heretofore, been proposed to add inorganic pigments to cellulosic spinning solutions with a view of forming therefrom dyed products. However, until the present time, it has been impossible to incorporate organic dyes into strongly alkaline spinning solutions without impairing the colors of the finished products. Viscose rayon, for example, is produced by treating cellulose with strong caustic alkali to form alkali cellulose which, subsequently, is transformed into cellulose xanthogenate (viscose) with the assistance of carbon bisulphide. After ripening the viscose solution, it is spun into a sulphuric acid bath, etc., to form regenerated cellulose. The sulphur is removed from the regenerated cellulose with alkaline sulphides, such as ammonium sulphide, etc. Finally the viscose products are bleached with sodium hypochlorite solutions, etc.

Cuprammonium cellulose is produced by dissolving cellulose in copper hydroxide-ammonia solution and spinning the dissolved cellulose into acid or alkaline spinning baths. The products are then freed from copper and bleached. In a similar manner, artificial products are made from alkaline solutions of alginates, silk fibroin, gelatine, etc. From the above, it will readily be recognized that only such organic dyes can be incorporated into alkaline spinning baths which are resistant to acids, desulphurizing agents and bleaching compounds, otherwise satisfactory products cannot be obtained. It is for this reason that rayon products have, heretofore, been exclusively dyed in finished form, i. e., after removal of sulphur, bleaching agents, etc. Unexpectedly, I have found that a number of organic dyes, for example "Triazol-Echtrot C (Griesheim)", "Toluylenrot (Griesheim)", "Triazol-Reinblau R", etc. can be admixed with strongly alkaline spinning solutions without being decomposed by the same. These dyes are manufactured by the "Chemische Fabrik Griesheim-Elektron" in Germany and sold under the aforementioned tradenames.

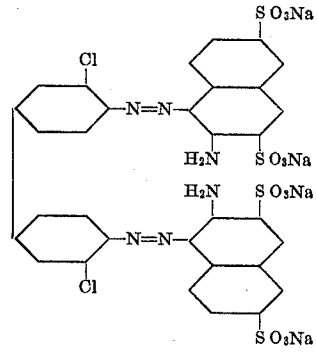
These dyestuffs have the following chemical structures:

(1) Triazol-Echtrot C (Griesheim): $C_{26}H_{19}N_5O_7SNa_2$



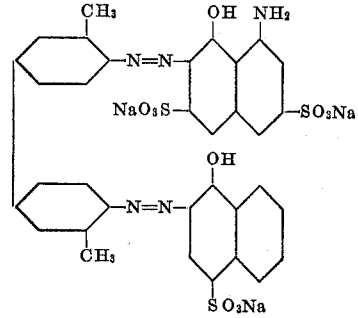
G. Schultz "Farbstofftabellen", VII edition, Vol. I, pp. 171, No. 410.

(2) Toluylenrot (Griesheim): $C_{32}H_{18}N_6O_{12}S_4Na_4Cl_2$



G. Schultz "Farbstofftabellen", VII edition, Vol. I, pp. 177, No. 425.

(3) Triazol-Reinblau R (Griesheim): $C_{34}H_{24}N_6O_{11}S_3Na_3$



G. Schultz "Farbstofftabellen", VII edition, Vol. I, pp. 193, No. 469.

They may be dissolved and/or dispersed in alkaline spinning solutions to form therewith after extrusion uniformly dyed products. The proportions of dye, added to the spinning solutions, may

be varied at will in order to produce any desired color-shade, and it is possible to admix a plurality of different dyes with a single batch of spinning solution. Instead of admixing the dyes with the whole bulk of spinning solution before entering the spinning machines, it is advisable to strongly dye a portion of the spinning solution and to mix it with the rest of spinning solution just before entering the spinning machines.

Modifications of my invention will readily be recognized by those skilled in the art, and I wish to include all modifications falling within the scope of the appended claims.

I claim:

1. A spinning solution for the manufacture of dyed artificial products comprising a viscose solution and a dye of the group consisting of "Triazol-Echtrot C (Griesheim)" having the formula $C_{29}H_{19}N_5O_7SNa_2$, "Toluylenrot (Griesheim)" having the formula $C_{32}H_{18}N_6O_{12}S_4Na_4Cl_2$ and "Triazol-Reinblau R" having the formula

$C_{34}H_{24}N_5O_{11}S_3Na_3$.

2. A spinning solution for the manufacture of dyed artificial products comprising a cellulosic solution of the group consisting of viscose and cuprammonium cellulose and an organic dyestuff of the group consisting of "Triazol-Echtrot C (Griesheim)" having the formula $C_{29}H_{19}N_5O_7SNa_2$, "Toluylenrot (Griesheim)" having the formula $C_{32}H_{18}N_6O_{12}S_4Na_4Cl_2$ and "Triazol-Reinblau R (Griesheim)" having the formula $C_{34}H_{24}N_5O_{11}S_3Na_3$.

3. A spinning solution for the manufacture of dyed artificial products comprising a cuproammonium cellulose solution and a dye of the group consisting of "Triazol-Echtrot C (Griesheim)" having the formula $C_{29}H_{19}N_5O_7SNa_2$, "Toluylenrot (Griesheim)" having the formula $C_{32}H_{18}N_6O_{12}S_4Na_4Cl_2$ and "Triazol-Reinblau R" having the formula $C_{34}H_{24}N_5O_{11}S_3Na_3$.

RUDOLPH S. BLEY.