

## UNITED STATES PATENT OFFICE

2,148,655

## COLORATION OF TEXTILE MATERIALS

George W. Seymour, Cumberland, Md., assignor  
to Celanese Corporation of America, a corpora-  
tion of Delaware

No Drawing. Application September 29, 1937,  
Serial No. 166,328

9 Claims. (Cl. 8-61)

This invention relates to improvements in the treatment of textile and other materials and more especially to the treatment of textile materials containing an organic derivative of cellulose to render colorations thereon faster to fading.

An object of this invention is the economic and expeditious production of improved dyed textile and other materials containing an organic derivative of cellulose. Another object of this invention is the treatment either before, during or after dyeing of a fabric containing an organic derivative of cellulose in such a manner as to render the fabric faster to light, acid and perspiration fading. A still further object of this invention is the production of compounds which when added to an organic derivative of cellulose render the same, when dyed, faster to light, acid and perspiration fading. Other objects of the invention will appear from the following detailed description.

It has been found that many dyed textile materials that contain an organic derivative of cellulose are subject to light and acid fading accompanied by the development of less stable materials, that is, the filaments or fibers of the yarn become weaker, have a lower melting point, and a lower viscosity when dissolved in a solvent. Many expedients have been used to correct this undesirable property, such as physical and/or chemical stabilizing treatments of the organic derivatives of cellulose prior to forming it into filaments or fibers. These treatments have greatly improved the stability of the materials especially when they are not dyed. When such materials are dyed or given other finishing treatments the stability thereof is decreased and continues to decrease with age and exposure. The decrease in stability of the materials, when they are dyed with certain anthraquinone dyes, is accompanied by a change in the color of the material. I have now found that if the textile materials are treated with a morpholine compound not only the stability of the organic derivative of cellulose is greatly improved but also the fastness of color of the dyed materials is enhanced. These improvements are obtained whether the color is imparted thereto by anthraquinone dyes or other types of dyes.

In accordance with this invention, fabrics or other materials such as filaments, yarns, fibers, films, foils, etc., containing an organic derivative of cellulose are treated with a derivative of morpholine such as phenylene dimorpholine, phenyl morpholine, phenyl amino morpholine and para-

hydroxy phenyl morpholine. For the purpose of describing this invention and in the claims such compounds are referred to broadly as phenyl morpholines. This treatment is given the material to effect a stability of the organic derivative of cellulose present and to improve the fastness of colors imparted thereto by dyestuffs. The treatment is preferably given the material during a dyeing operation, but may also be given as a separate operation or in connection with any other operation either before or after the dyeing operation.

The fabrics that most particularly lend themselves to this invention are those formed substantially wholly of filaments or fibers of organic derivatives of cellulose. However, fabrics of mixed yarns containing both filaments or fibers of organic derivatives of cellulose and other filaments or fibers, such as silk, cotton, wool, etc., may be treated and also fabrics made wholly of materials other than an organic derivative of cellulose. The organic derivative of cellulose may be an organic ester of cellulose or a cellulose ether or a mixed ether or ester of cellulose. Examples of the organic esters of cellulose are cellulose acetate, cellulose formate, cellulose propionate and cellulose butyrate, while examples of the cellulose ethers are ethyl cellulose, methyl cellulose and benzyl cellulose.

The fabric to be treated may be formed by weaving, warp-knitting, circular-knitting, knotting or netting of yarns of any degree of twist. Although this invention is primarily applicable to fabrics, it may in an obvious manner be applied to yarns, straws, monofilaments, films, foils, etc. in packages such as hanks, spools, bags or rolls, or during any winding operation. The filaments, fibers or yarns of organic derivatives of cellulose may contain, besides the cellulose derivatives, effect materials such as pigments, filling materials, dyes or lakes, fire retardants, plasticizers and lubricants. The reagents necessary to produce the desired properties obtained through the use of the effect materials are known to one practicing the art. This invention is of particular importance in connection with organic derivatives of cellulose materials that contain metal oxide pigments such as titanium oxide or zinc oxide.

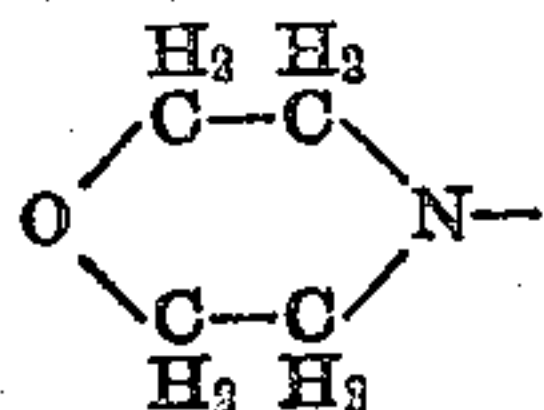
The organic derivative of cellulose base material may be made by any suitable method and, prior to its being formed into fibers or filaments, it may be treated with water or steam, with or without pressure, or given other types of stabilizing treatments. The fibers or filaments may be formed from a solution of the organic deriva-



tive of cellulose by either the wet or dry method of forming filaments, and these fibers or filaments may be coated or otherwise treated with lubricants, sizes, etc., that may be necessary for their

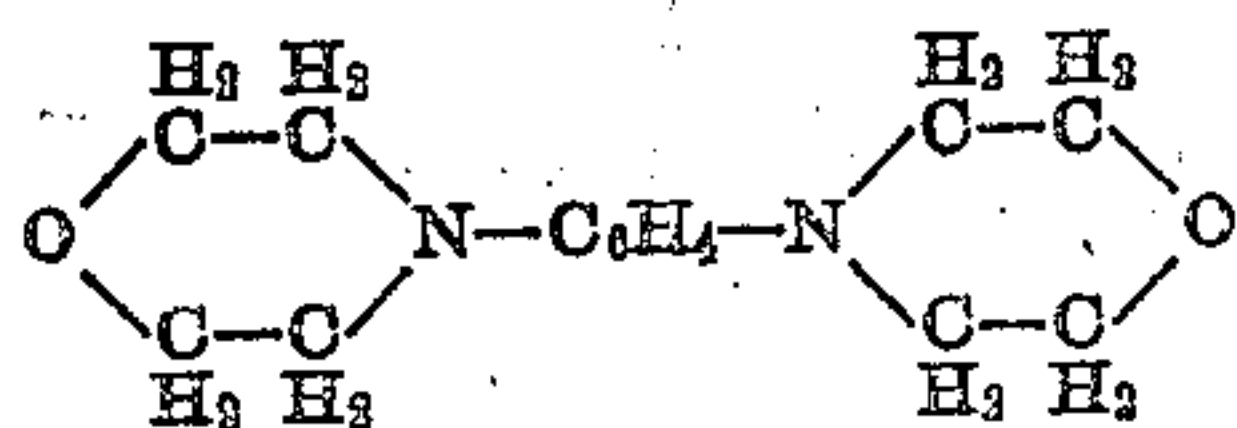
production into textile materials such as yarns and fabrics. The fibers or filaments may be processed in the usual manner to yarns and fabrics.

The fabrics or other materials are treated with a morpholine compound, that is, a compound containing the radicle

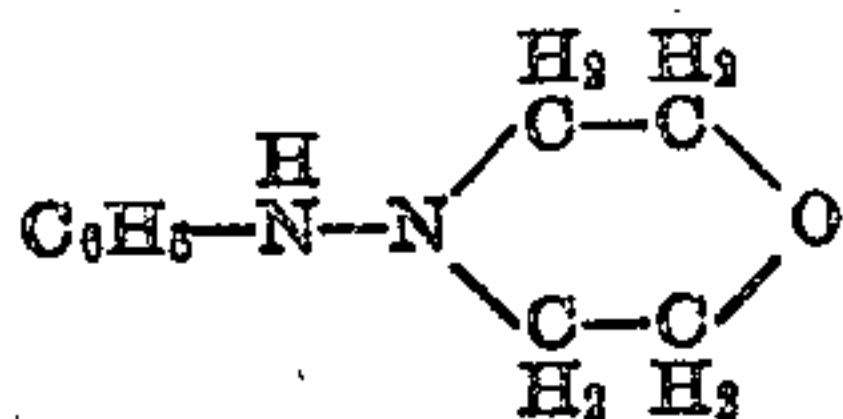


or derivatives thereof formed by a substitution of one or more of the hydrogens. Examples of morpholine compounds and their ascribed formulas are:

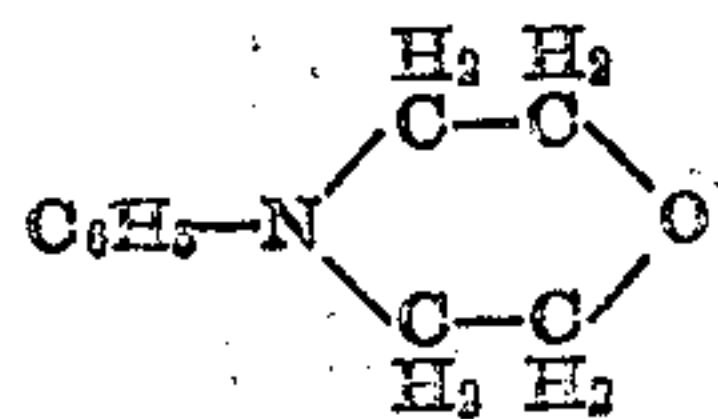
Phenylene dimorpholine



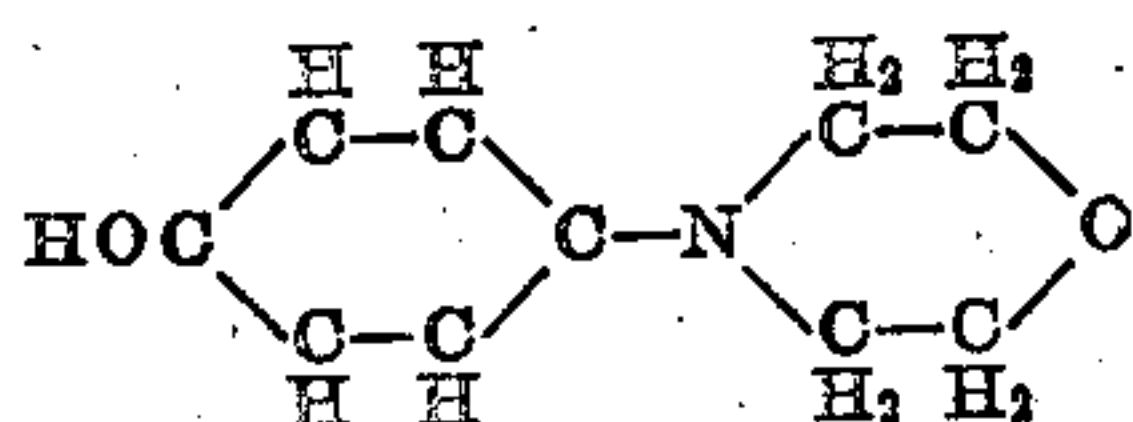
Phenylamino morpholine



Phenyl morpholine



Parahydroxy phenyl morpholine



These morpholine compounds may be produced by refluxing B=B dichloroethyl ether, an amine and sodium or potassium hydroxide, say about 40% strength, for about 3 to 6 hours. The product may be washed with alcohol and purified by recrystallizing from benzene. For instance, phenylene dimorpholine may be formed by refluxing B=B dichloroethyl ether, p-phenylene diamine and potassium hydroxide, while phenylamino morpholine may be formed from B=B dichloroethyl ether and phenyl hydrazine, and phenyl morpholine may be formed from B=B dichloroethyl ether and aniline.

Solutions, emulsions and dispersions of the morpholine compounds of a strength suitable for application to the textile or other material may be directly prepared. If desired, however, the compounds may first be brought into the form of solid, powder, paste, liquid or other more or less concentrated preparations, capable of yielding, by dilution with water, soap solution or other liquid, treating baths containing the morpholine compounds in suitably distributed form and in suitable concentration. Pastes and powders containing the morpholine compound may be formed by grinding or mixing said compound with dispersing agents and/or protective colloids with or without water. Where the morpholine compound is to be applied to textile materials in connection with a dyeing operation it may be ground or

mixed with the dye, dispersing agents and detergents. The morpholine compound may be mixed into the commercial dye pastes and powders whereby upon adding these to water to form a dye bath there is also formed the morpholine compound treating bath. The morpholine compound may also be added to printing pastes or powdered dyes to be used in printing pastes. Compositions of matter containing the morpholine compound together with dyestuffs and particularly dyestuffs of the anthraquinone series containing free amino or alkyl amino groups also form part of the present invention. These compositions of matter may be of a type to be applied directly for the coloration of materials in fast shades as in printing or of a type to be diluted with or dispersed in water for the coloration of materials in fast shades as in dyeing.

A further method of treating textile or other materials with the morpholine compound is to add the said compound to lubricants, sizes, etc., and apply it to the materials during a winding operation by means of wicks, rollers or other furnishing devices. For instance, a lubricant dressing containing a small amount of a morpholine compound and sulphonated olive or peanut oil, with or without mineral oil, vegetable oil, and/or animal oil, may be applied to a cellulose acetate yarn as it leaves the spinning cabinet in which it is formed. The amount of morpholine compound employed should be sufficient so that there is applied to the yarn from 0.01 to 3% based on the weight of the yarn. Also, in treating artificial yarns or filaments the morpholine compound may be added to the spinning solution from which they are formed. A still further method of treating the textile materials with the morpholine compound is by means of a bath containing said compound after the dyeing and scouring operation.

The amount of morpholine compound employed is preferably from 1 to 3% based upon the weight of the material to be treated although lesser or greater amounts may be employed. In operations wherein the morpholine compound is applied to the material with lubricants, sizes, etc., by means of furnishing devices such as wicks, rollers, slashers, padding mangles, etc., it may be necessary to continually add to the treating composition an additional amount of the morpholine compound so that the material being treated acquires a uniform amount the same.

When the morpholine compound is applied in the form of a bath to dyed textile materials the bath may also contain from 0.05 to 0.3 gram per litre of lime or other oxide, hydroxide or carbonate of an earth-alkali metal.

As illustrations of this invention and not as limitations, the following examples are given:

#### Example I

Textile material consisting of cellulose acetate is dyed in a dye bath of thirty times its weight and a temperature of 80° C., said dye bath containing an anthraquinone dyestuff having an affinity for cellulose acetate and 2% on the weight of the textile material of phenylene dimorpholine. The material after being dyed to the desired shade is separated from the dye bath, rinsed, scoured in a soap solution and again rinsed. The material is then dried in the customary manner. The textile material is found to be greatly improved in stability insofar as any change in the cellulose acetate is concerned, and there is also found to be a great improvement in



the fastness to light and acid fading of the color imparted thereto.

#### Example II

5 To a cellulose acetate yarn, as it leaves the spinning cabinet in which it was formed and while it still contains some residual solvent, is applied by means of a furnishing roller a lubricating dressing containing sulphonated olive oil, oxidized olive oil, mineral oil and phenylene dimorpholine.  
10 The lubricant dressing contains about 5% of the morpholine compound and about a 2% by weight dressing is applied. The yarn is hanked and dyed with an anthraquinone dyestuff. The yarn after scouring and drying is found to be greatly im-  
15 proved in stability of both the yarn and color imparted thereto than a blank run from the same charge of cellulose acetate.

#### Example III

20 The same procedure is followed as in Example I except that the final rinse after dyeing contains about 1 gram per litre of calcium hydroxide.

It is to be understood that the foregoing specification is given as a description and that many  
25 modifications may be made therein without departing from the spirit of my invention.

Having described my invention, what I desire to secure by Letters Patent is:

1. Process for producing fast colorations on  
30 textile materials having a basis of organic derivatives of cellulose, which comprises incorporating a substantially colorless phenyl morpholine in the materials and dyeing the materials with an anthraquinone dyestuff which has affinity for the  
35 materials and which contains a substituent radicle selected from the group consisting of the unsubstituted amino and alkylamino radicles.

2. Process for producing fast colorations on textile materials having a basis of cellulose acetate, which comprises incorporating phenylene dimorpholine in the materials and dyeing the materials with an anthraquinone dyestuff which has  
40 affinity for the materials and which contains a substituent radicle selected from the group consisting of the unsubstituted amino and alkyl-  
45 amino radicles.

3. Process for producing fast colorations on textile materials having a basis of cellulose acetate, which comprises incorporating phenylamino  
50 morpholine in the materials and dyeing the materials with an anthraquinone dyestuff which has affinity for the materials and which contains a substituent radicle selected from the group

consisting of the unsubstituted amino and alkylamino radicles.

4. Process for producing fast colorations on textile materials having a basis of cellulose acetate, which comprises incorporating in the materials up to 3% of their weight of a substantially  
5 colorless phenyl morpholine and dyeing the materials with an anthraquinone dyestuff which has affinity for the materials and which contains a substituent radicle selected from the group consisting of the unsubstituted amino and alkylamino  
10 radicles.

5. Textile materials which have a basis of cellulose acetate and which contain a substantially colorless phenyl morpholine, dyed with an anthraquinone dyestuff which has affinity for cellulose acetate materials and which contains a substituent radicle selected from the group consisting of the unsubstituted amino and alkylamino  
15 radicles.

6. Textile materials which have a basis of cellulose acetate and which contain phenylene dimorpholine, dyed with an anthraquinone dyestuff which has affinity for cellulose acetate materials and which contains a substituent radicle selected from the group consisting of the unsubstituted  
20 amino and alkylamino radicles.

7. Textile materials which have a basis of cellulose acetate and which contain phenylamino morpholine, dyed with an anthraquinone dyestuff which has affinity for cellulose acetate materials and which contains a substituent radicle selected from the group consisting of the unsubstituted  
25 amino and alkylamino radicles.

8. Textile materials which have a basis of cellulose acetate and which contain up to 3% of their weight of a substantially colorless phenyl morpholine, dyed with an anthraquinone dyestuff which has affinity for cellulose acetate materials and which contains a substituent radicle selected from the group consisting of the unsubstituted  
30 amino and alkylamino radicles.

9. Textile materials which have a basis of cellulose acetate, which contain titanium dioxide, and which contain a proportion up to 3% of their weight of a substantially colorless phenyl morpholine, dyed with an anthraquinone dyestuff which has affinity for cellulose acetate materials and which contains a substituent radicle selected from the group consisting of the unsubstituted  
35 amino and alkylamino radicles.

GEORGE W. SEYMOUR.