

# UNITED STATES PATENT OFFICE

2,635,969

## PHOSPHORESCENT YARNS AND METHOD FOR PRODUCING SAME

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No Drawing. Application March 3, 1950,  
Serial No. 147,579

10 Claims. (Cl. 117—33.5)

1

The present invention relates to a method of imparting luminescence in particular phosphorescence to threads and yarns made of natural and synthetic fibers particularly spun yarns such as wool, cotton, silk, nylons, acetate rayon and combinations of the same, except that where the melting point of the synthetic fibers is low as for instance lower than the boiling point of water, the methods of the present invention may not be wholly successful.

The present invention particularly relates to applying phosphorescent crystals to nylon fibers and yarns in such a manner that the physical characteristics of the yarn remain unchanged particularly in regard to softness, flexibility, general appearance and texture. For the most part in the prior art, phosphorescence or luminescence has been applied to yarns and fibers by subjecting the yarns and fibers, first to treatment with phenol, thymol, or some other equivalent substances and then adhering a luminous compound thereto. It is obvious that with such treatment yarns or fibers will become somewhat stiff and have their character considerably changed due to the fact that the twisted fibers become joined and therefore immobilized and also to the fact that they become coated or partially coated. This destroys to some extent both the flexibility of the yarn, and also the soft texture and particular sheen which it is desired to maintain in silks and rayons.

Other methods of treating texture fibers and yarns have been to impregnate and coat the yarns with some thermoplastic materials for the purpose of imparting strength to them and in some cases for the purpose of binding the yarns either to cross yarns as in knitted and woven fabrics or for use as a filler or to decrease the porosity of the yarns and the fabrics. It is readily seen that these methods are not readily adaptable for the purpose of the present invention.

In the present invention the phosphorescence is imparted by suitable crystals of activated metallic salts such as zinc sulphide or sulphide of calcium, strontium, cadmium, barium, magnesium, and the like, the choice depending upon the compatibility of these crystal solids in the medium in which they are used as will be set forth clearly below. These crystals should be of a particularly small size such for instance that they will pass through a 90-325 mesh screen. The crystals are suspended in a suitable liquid

2

vehicle or lacquer in which the yarns are dipped or passed through whereby the crystals are deposited in such a manner that they will permanently remain with the yarns. The vehicle or lacquer as has been previously stated must not change the texture of the yarns. The applicant has found that which a variety of liquid baths may be used for this purpose, the best results are obtained by those in which the ratio of the plasticizer to the resin used is rather large. While ratios as low as 4-1 of the plasticizer to the resin may be found to be satisfactory this would appear to be the lower limit and better results are obtained from ratios ranging between 8-1 and approximately 20-1 are more satisfactory. The applicant has found that when the resin content is relatively high, that is when the ratio above is low, there is a considerable tendency for the yarns to become stiffer, and it further appears that the resin does not penetrate the yarn but rather coats the yarn thereby imparting a characteristic of the resin itself rather than permitting the character of the yarn to prevail. On the other hand where the ratio is greater, the liquid vehicle appears to carry the phosphorescent crystals into intimate contact with the fibers penetrating the fiber structure and becoming imbedded quite completely in the interstices between the twisted fibers. Further than this when the yarns are dried out, there is no residue of resin left on the fibers and the fibers have their original characteristics of softness, flexibility, texture and sheen that were the properties of the original materials. In other words, nylon and silk have the appearance of nylon and silk except for the phosphorescent properties which are noticeably visible and impart radiance to the yarn in the dark after previous activation by light.

The applicant has found the following method to be satisfactory. A relatively small quantity of such a resin as butyl methacrylate is dissolved in substantially equal quantities of tricresylphosphate and olive oil to which zinc sulphide crystals are added in quantities sufficient to be completely thoroughly distributed throughout the whole solution. The solution should be stirred while heating to approximately 100°-150° C., the range being dependent upon the character of the resin and the temperature which the yarn will withstand. The yarns may then be immersed in the bath and kept there for sufficient time to permit them to become thoroughly wetted

3

and the liquid to thoroughly penetrate the fibers. The time may vary considerably but usually is not more than five minutes and may be approximately one to one and a half minutes. If the yarn is in a single strand a continuous process may be used by simply drawing the strand through the liquid. The yarn is then washed hot directly from the bath if desired although some cooling may be permitted to remove the excess of resin, and then the yarns are washed with hot soap or equivalent detergent and water after which they are dried.

I have found specifically the following formula to be satisfactory:

	Parts by weight
Tricresylphosphate .....	7.2
Olive oil .....	7
Butylmethacrylate dissolved in ketones .....	.6
Zinc sulphide crystals, 200 mesh .....	2

The ratio of plasticizer to resin in this case is about 24-1. The method of mixing, heating and washing is the same as previously described.

The following formula has also proved to be satisfactory:

	Parts
Dibutylphthalate .....	4.9
Dimethylhexylphthalate .....	3.9
Methylmethacrylate .....	.6
Ethylacetate .....	5.9
Zinc sulphide crystals .....	2

The same process should be followed in applying this formula as in that described above. The ratio of the plasticizer to resin in this case is 14.6 to 1.

Another formula which has also been found successful comprises:

	Parts
Dibutylphthalate .....	4.9
Olive oil .....	3.2
Polyethylene .5 dissolved in an acetate solvent commonly known as DYLT.	5
Zinc sulphide crystals .....	2

"DYLT" is a trade name given by a manufacturer for a liquid comprising polyethylene dissolved in acetone.

The ratio of plasticizer to resin in this case is about 16-1.

Other resinous compounds which may be used comprise polystyrene resin, cellulose acetate, polyvinyl, polyethylene, acrylic resins and mixtures and co-polymers of the same. An example with the use of polystyrene resin is as follows:

3.9 parts di-2-ethylhexylphthalate, commonly known by the trade name D. O. P.

4.9 parts di-butylphthalate

1 part polystyrene resin dissolved in the necessary amount of acetone, toluol, methylethylketone or other ketones.

2 parts zinc sulphide crystals or other compatible crystals.

The ratio of plasticizer to resins in this case is about 8-1.

In all cases the mixture is heated to a temperature of 100° to 150° C. with the crystals being stirred thoroughly into the solution. The temperature of the liquid bath or solution must be low enough so as not to affect the characteristics of the yarns that are used. If such yarns as rayons are used, the temperature of the bath must be considerably lower than when nylons are used and should be subjected to heating for a shorter time. While the yarns are still warm and preferably hot, the excess of the bath is washed off in alcohol or carbon tetrachloride or

4

other solvents and finally the yarns are washed with soap and warm water.

I have found that in the methods described above the liquids readily and quickly penetrate within and between the yarn fibers in a homogeneous and uniform manner and do not form a noticeable film on the yarn. Under microscopic examinations with high power magnification, it is difficult to tell a treated yarn from an untreated yarn. The crystals being dispersed uniformly and over the whole yarn, the luminous response is likewise uniform and even from all parts of the visible yarn. Such yarns may be used in the knitting of stockings to produce a desired pattern, or they may be used to seam the yarn as for instance the back seam, or they may be sewed into the stocking for special patterns and designs.

The yarn of the present invention may also be applied to woolsens or to other synthetic fiber compositions and may be usefully employed for designs such as rugs or carpets in clothing and for other similar purposes.

The invention, however, finds special utility in its application to nylons, not only to the light yarns used in the manufacture of sheer hosiery, but also in heavier yarns and in seaming yarns which are used to sew up full fashioned ladies' hose.

Having now described my invention, I claim:

1. A method of imparting phosphorescence to spun yarns which comprises preparing a liquid mixture, said liquid mixture containing a resin selected from the group consisting of polystyrene, polyvinyl, polyethylene, acrylic resin and copolymers thereof, the resin being dissolved in an organic solvent, said liquid mixture containing a plasticizer compatible with the resin and phosphorescent crystals of a size which will pass through a screen of 20 to 325 mesh dispersed therein, the ratio of plasticizer to resin being between 4:1 and 24:1; heating the liquid mixture to between 100° C. and 150° C., immersing the yarns in the liquid mixture for a time sufficient to have the yarns become thoroughly wetted, removing the yarns from the liquid, removing excess liquid from the yarns and then washing the yarns and drying the same.

2. The method of claim 1 in which the liquid mixture contains:

	Parts
Tricresyl phosphate .....	7.2
Olive oil .....	7
Butyl methacrylate .....	.6
Zinc sulphide crystals .....	2

3. A spun yarn provided with a phosphorescent coating within the interstices of the fibers thereof, said yarn being produced by the method of claim 1.

4. The method as in claim 1 where the yarns comprise nylon spun yarns.

5. The method as in claim 1 where the yarns comprise natural spun silk.

6. In a method of making spun yarn phosphorescent the step of wetting the yarn thoroughly in a liquid composed substantially as follows:

	Parts
Dibutylphthalate .....	4.9
Dimethylhexylphthalate .....	3.9
Methylmethacrylate .....	.6
Dissolved in ethylacetate .....	5.9
Phosphorescent crystals, 90 to 325 screen mesh size .....	2.00

5

7. In a method of making spun yarn phosphorescent the step of wetting the yarn thoroughly in a liquid composed substantially as follows:

	Parts	5
Dibutylphthalate .....	4.9	
Dimethylhexylphthalate .....	3.9	
Methylmethacrylate .....	.6	
Dissolved in ethylacetate.....	5.9	
Zinc sulphide crystals, 90 to 325 mesh ----	2.00	10

8. In a method of making spun yarn phosphorescent the step of wetting the yarn thoroughly in a liquid composed substantially as follows:

	Parts	15
Dibutylphthalate .....	4.9	
Olive oil .....	3.2	
Polyethylene -- .5 dissolved in an acetate solvent		
Zinc sulphide crystals .....	2	20

6

9. A method as in claim 1 where the yarn comprises natural spun cotton.

10. A method as in claim 1 where the yarn comprises natural spun wool.

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