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3,137,655

## OPTICAL BRIGHTENING

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5 Claims. (Cl. 252—301.2)

This invention relates to the optical brightening of polyamide textiles, and in particular to the use of a novel brightening agent having improved properties of wash fastness and light fastness when incorporated in polyamides.

It is known that synthetic linear polyamides such as nylon and polycaprolactam become yellow as they age, and this quality is especially noticeable and undesirable in textile materials which are white rather than colored. In order to avoid or reduce this yellowing, it has been a common practice to incorporate so-called optical brightening agents in the polyamide textile material, particularly those brightening or whitening agents which give a more or less intense blue fluorescence, thereby masking the yellow color of the polyamide. Many compounds have been employed for this purpose, and the whitening or brightening effect is often quite successful for a short period of time after treatment of the textile. However, where the textile must be frequently washed, the brightening effect wears away because of a poor wash fastness of the brightening additive. Also, the light fastness of the brightening agent is often poor because many of these compounds will decompose under the action of light, and the decomposition products which adhere to the textile cause an even greater than normal yellowing effect. Permanent brightening effects are therefore quite difficult to achieve, and it is usually necessary to add fresh agents to a textile material at frequent intervals.

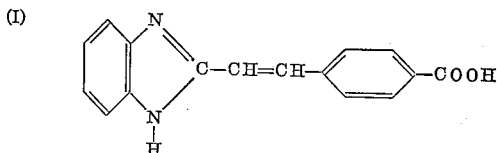
One object of the present invention is to provide an optical brightening agent for polyamide textiles which is characterized by its outstanding qualities of wash fastness and light fastness.

Another object of the invention is to provide a method of treating polyamide textiles such that they will exhibit an enduring brightness in spite of repeated washing and exposure to light.

Still another object of the invention is the provision of an improved polyamide textile as a composition of matter containing the special optical brightening agent disclosed hereinafter.

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

In accordance with the present invention, it has now been found that polyamide textiles can be brightened or whitened so as to obtain the above objects by employing as the optical brightening agent the compound of the formula:



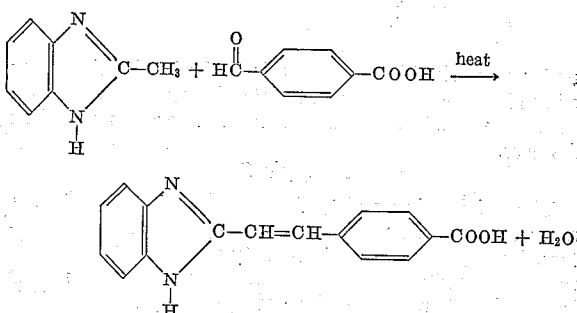
This compound is distinguished from previously known

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brightening agents by the presence of the carboxyl group in the benzimidazole derivative. It was quite surprising to find that this substitution of a carboxyl group would lead to an improved and lasting fluorescence and brightening effect because there has been a prevailing opinion in this art that the use of a carboxyl substituent, as well as chlorine and bromine substituent, will impair or destroy the desired fluorescence. Accordingly, the present invention is directly contrary to theoretical expectations, and no attempt will be made herein to give a scientific explanation of the results which have been obtained.

As polyamide textiles, the synthetic products known as nylon and polycaprolactam are especially improved by the optical brightening agent of the invention. The terms "textile" and "textile materials" are employed herein with reference to the solid polyamide regardless of the particular form which it may require for a certain textile use. The finished article may be in the form of filaments, fibers, yarn, thread, rope, woven or knitted fabrics, film, felted fibers or the like.

The brightening agent of the above formula can be readily obtained by reacting 2-methyl-benzimidazole with benzaldehyde-carboxylic acid-4 in the molten state according to the equation:



Thus, a mixture of about one mol of 2-methyl-benzimidazole to one mol of benzaldehyde-carboxylic acid-4 is simply heated until it melts, and the molten reactants are then further heated at the temperature of the melt for removal of water until the reaction is completed as evidenced by the solidification of the melt. The solid product can then be washed several times with cold alcohol to obtain the substantially pure brightening agent according to this invention.

The best brightening effect is obtained if the compound of the invention is incorporated in the polyamide by addition to the monomeric materials from which the polyamide is produced, e.g. by polycondensation. The addition of the brightening agent prior to or during polycondensation is therefore of special technical interest. In this way, the brightening agent is uniformly dispersed throughout the polyamide structure which can then be spun or extruded into various textile forms in the usual manner. If desired, the brightening agent can also be applied to the polyamide textile materials as an after-treatment, i.e. after polycondensation.

A good brightening or whitening effect can be achieved by employing at least 0.02% by weight of the brightening agent with reference to the weight of the polyamide. Large amounts of the brightening agent are possible but unnecessary, and the maximum amount should ordinarily not exceed about 0.5% by weight with respect to the polyamide. Optimum results have been obtained by em-

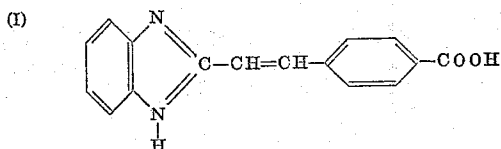
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playing about 0.1 to 0.2% by weight of the brightening agent in the finished article.

The following examples are intended solely for the purpose of illustrating the invention in a preferred form, and the invention is not to be limited thereby. Percentages are by weight unless otherwise specified.

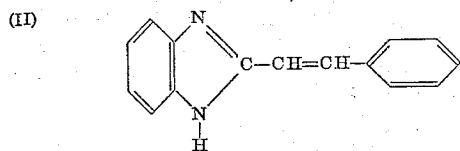
#### Example 1

The compound of the formula:



is added to an aqueous 95% caprolactam solution in an amount of about 0.1% (with reference to the finished polyamide). The monomer is polymerized in the usual manner, and the resulting polycaprolactam containing the designated brightening agent is melt-spun into filaments. These filaments exhibit a good luminescent or brightening effect which is not lost even after frequent washing. Under continuous exposure to light, the filaments do not become yellow and there is no indication of decomposition of the brightening agent or loss of its luminescent effect. As regards such light fastness, the brightening agent of the invention is superior to all known additives which are commercially available at the present time.

By comparison of the compound of the formula:



under otherwise identical conditions, the brightening agent of the invention provides a substantial and surprising improvement, both with respect to wash fastness and light fastness.

#### Example 2

The brightening agent of Example 1 is added in the same manner prior to polycondensation of the caprolactam, but in an amount of 0.2% by weight with reference to the finished polyamide article. The resulting product is likewise characterized by a very strong and enduring fastness to washing and light.

#### Example 3

The brightening agent of Formula I above is added to hexamethylenediamine adipate as the monomeric reactant in the production of nylon in an amount corresponding

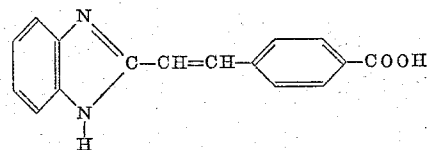
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to about 0.15% by weight with reference to the polyamide product. After polymerization, the polyamide is spun into nylon filaments by conventional melt-spinning. These filaments can be employed as continuous or staple filament yarns and woven or knitted to form fabrics or the like. The filaments and fabrics formed therefrom exhibit excellent light-fastness with a high stability to frequent washing.

This is a continuation-in-part application of our prior copending application Serial No. 13,993, filed March 10, 1960, now abandoned.

The invention is hereby claimed as follows:

1. A textile product comprising a polyamide selected from the group consisting of polycaprolactam and polyhexamethylenediamine adipamide containing as an optical brightening agent the compound of the formula:

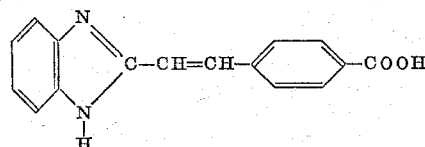


2. The textile product as claimed in claim 1 wherein the polyamide contains at least 0.02% by weight of the brightening agent.

3. The textile product as claimed in claim 1 wherein said polyamide has uniformly dispersed therein from 0.02% up to about 0.5% by weight of said optical brightening agent.

4. The textile product as claimed in claim 1 wherein said polyamide has uniformly dispersed therein from about 0.1% to 0.2% by weight of said optical brightening agent.

5. A synthetic linear polyamide textile article containing as an optical brightening agent the compound of the formula:



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