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(54) **Pigmented stain resistant nylon fiber.**

(57) Nylon carpet fibers are provided which have excellent resistance to being stained by acid dye food colorants. The fibers are prepared from pigmented nylon polymer that has been modified to contain aromatic sulfonated units as an integral part of its polymer chain.

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PIGMENTED STAIN RESISTANT NYLON FIBER

BACKGROUND OF THE INVENTION

5 1. Field of the Invention

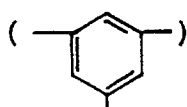
This invention relates to pigmented nylon fibers which have excellent stain resistance and resistance to oxidizing agents and to carpets containing such fibers. The term "fibers", as used herein, includes fibers of extreme or indefinite length (i.e. filaments) and fibers of short length (i.e. staple). The term "stain resistance", when used herein, means resistance to being stained by acid dye food colorants (e.g. FD&C Red Dye No. 40). The term "pigmented", when used herein with reference to nylon fibers, means nylon fibers containing compounds which are added to the nylon prior to fiber formation and which impart color to the fibers. The term "yarn", when used herein, means a continuous strand of fibers (i.e. singles yarn) or two or more continuous strands of fibers twisted together (i.e. plied yarn).

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2. Description of Prior Art

It is known that the fiber manufacture can improve the stain resistance of nylon carpet fibers by either coating the fibers during their manufacture with certain sulfonated condensation products (see U.S. 4,680,212) or by modifying the nylon polymer from which the fibers are made to contain aromatic sulfonated units (e.g.)

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SO₃M (M is an alkali metal)

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as an integral part of its chain (see U.S. 4,579,762). Unfortunately, the sulfonated condensation product coating makes the coated fibers more difficult to dye with acid dyes by conventional techniques and fibers made from the modified nylon polymer can not be dyed with acid dyes by conventional techniques.

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SUMMARY OF THE INVENTION

The present invention provides nylon fibers which have excellent stain resistance for extended periods of time and do not suffer from the dyeability difficulties of the above-mentioned prior art fibers. The fibers comprise a modified nylon polymer containing a pigment dispersed therein. The polymer is modified in that it contains aromatic sulfonated units as an integral part of its polymer chain. The amount of the sulfonated units present in the polymer chain is selected to provide fibers which, when subjected to the 8-Hour Red Dye No. 40 Staining Test (hereinafter described), show no visible staining. FD&C Red Dye No. 40 is one of the most commonly used acid dye colorants and also one which severely stains nylon at room temperature. This dye is found in beverages (e.g. cherry Kool Aid), gelatins and other common foods and beverages. In general the fibers of the invention are also resistant to strong oxidizing agents (bleaches), such as, benzoyl peroxide and sodium hypochlorite, coffee, red wine, and grape juice.

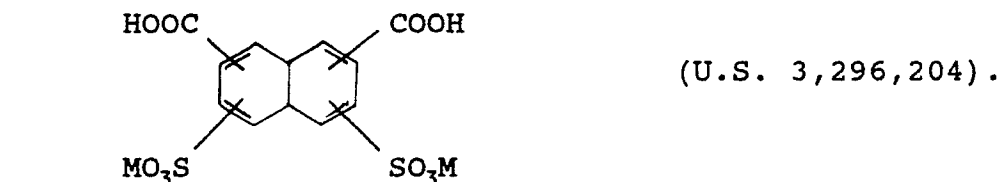
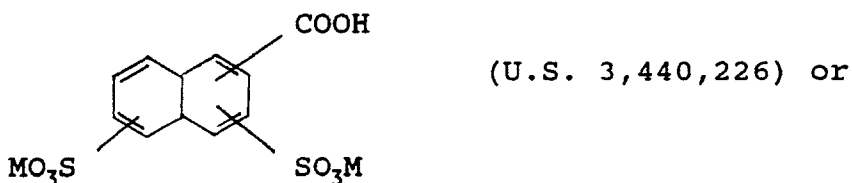
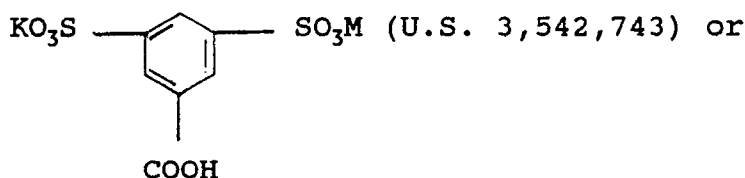
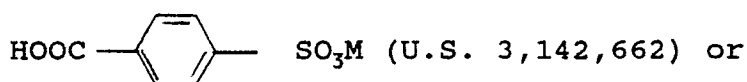
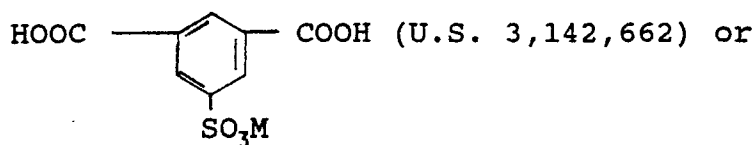
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Description of the Preferred Embodiments

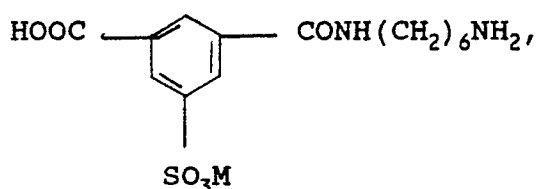
The fibers of the invention are particularly useful for making two-ply carpet yarns which may be used in a conventional manner in the construction of carpets. Such fiber (i.e. carpet fibers) will have a denier of at

least 6 and, typically, from 15 to 25.

Modified nylon 66 and nylon 6 polymers useful in practicing the present invention may be prepared by the techniques described in the prior art. According to these techniques the polymer is prepared by replacing a portion of the nylon-forming monomers with a corresponding molar amount of an appropriate sulfonated aromatic monomer. For example, in the case of nylon 66 the adipic acid monomer is replaced by

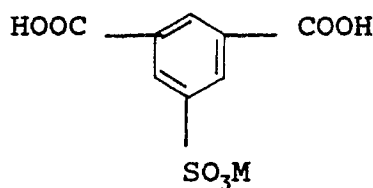


In the case of nylon 6, the polymer is similarly modified by adding an appropriate amount of a monomer, for example



to the caprolactam monomer.

The preferred aromatic sulfonated monomers useful in practicing the invention is chain-extending monomer of the formula



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where M is preferable sodium or potassium. Usually an effective amount of the sulfonated monomer will be in the range of 0.25 to 2.5 molar percent, based on the moles of nylon. While modified nylon 66 or nylon 6 polymers are preferred, other nylon polymers could be modified in a similar manner and also used to practice the invention.

Pigments useful in practicing the present invention are those which are stable under nylon melt-spinning conditions, that is, are capable of withstanding the chemical and thermal environment of molten nylon in an inert atmosphere and include one or more compounds conventionally used in making pigmented nylon fibers, such as, phthalocyanine copper complex (blue), lead sulfochromate (yellow), ferric oxide (red), zinc sulfide (white) phthalocyanine green, phthalocyanine blue, cadmium sulfide (yellow), cadmium selenide (red), carbon black, titanium dioxide. The pigment may be uniformly dispersed into the modified nylon fibers by conventional techniques, for example, by mixing a pigment composition with the molten modified nylon polymer from which the fibers are shaped or by mixing (blending) the composition with modified nylon flake that is then used to make the fibers by conventional melt-spinning techniques. Preferred pigment composition are those comprising pigment dispersed in nylon 6 in amounts, for example, ranging from 20 to 50 parts by weight of pigment and from 50 to 80 parts by weight of nylon 6. Such compositions are commercially available. Normally, from 1 to 5% by weight of pigment is used, based on the weight of modified nylon to be pigmented. Preferably, pigments used in practicing the invention are those that are also resistant to all of common household substances including bleaches and mustards.

The following examples are given to further illustrate the invention.

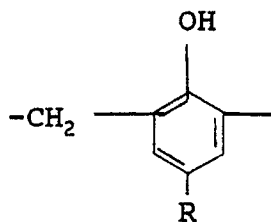
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Example 1

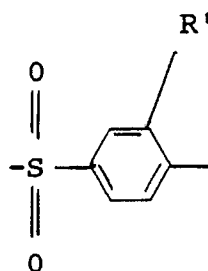
In this example the staining of cut pile tufted carpet incorporating pigmented modified nylon 66 fibers of the present invention is compared to two corresponding carpets, one incorporating unmodified nylon 66 fibers (Control A) and the other incorporating unmodified nylon 66 fibers coated with stainblocker (Control B).

Control A is made in the following way. A 300 filament, 60 denier per filament (dpf), nylon 66 yarn is prepared by extruding fiber-forming nylon 66 of commercial grade at a melt temperature of 282°C. downwardly through the orifices of a 300-hole spinneret into a conventional melt spinning chimney, measuring approximately 1.8 meters in length, to form a corresponding number of molten streams. The chimney is adapted to receive a cross-flow of cooling air at ambient temperature at a velocity of 270 meters/min. The molten streams solidify in the chimney to form filaments. The filaments are passed from the chimney through a conventional steam conditioning tube measuring about 1.2 meters in length where the filaments are treated with steam. The filaments are passed from the conditioning tube over a conventional metered finish applicator where an aqueous finish is applied and the filaments are converged to form a yarn. The yarn is then passed over and around a driven feed roll (450 meters/min.) and its associated separator roll with several wraps. The yarn is then collected on a bobbin under a slight tension to facilitate winding of the yarn onto the bobbin. The yarn is then unwound from the bobbin and combined with 54 like yarns to form a tow having a total denier of about 1,000,000. The tow is drawn over rolls to provide nominal 18 dpf tow, crimped in a conventional stuffer box and cut into 7-1/2 inch (19.05 cm) staple. The staple is carded, drafted, spun on a conventional ring spinning frame to provide a 3.75 cotton count singles yarns having about 5.25 tpi (206.5 tpm) of twist in the Z-direction. Two of these yarns are then plied with 4.75 tpi (186.4 tpm) of twist in the S-direction.

Control B is made in the same manner except that a sufficient amount of a stainblocker consisting of repeating units of the formula

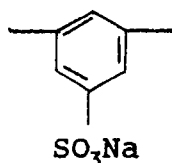


10 where R is



OH and R' is $-\text{SO}_3\text{Na}$ in at least 50% of the units and hydrogen in the remaining units is added to the aqueous finish to provide yarn coated with 3500 ppm of the stainblocker.

25 Pigmented modified nylon 66 yarn of the present invention is made in the same manner as Control A except that, instead of using nylon 66 polymer to prepare the fibers, a nylon 66 polymer modified to contain 13,943 ppm of the radical of the formula



35 as an integral part of its polymer chain (2300 ppm of sulfur) is used and 1%, based on the weight of modified nylon 66 polymer, of a composition comprising 25% by weight phthalocyanine green pigment dispersed in nylon 6 is added to (blended with) the molten modified nylon just prior to extrusion thereof. The modified polymer is prepared in a conventional manner by adding an appropriate amount of the sodium salt of 5-isosulfophthalic acid to nylon 66 salt prior to polymerization.

40 Each of the yarns is heatset using conventional Suessen heatsetting equipment and conditions. Each yarn is then tufted on a 1/8 inch (0.32 cm) gauge cut pile tufting machine into a primary backing using 8 stitches per inch (31.5 stitches per 10 cm). The pile height is 5/8 in. (1.6 cm) and 27 oz. of yarn is used per square yard of carpet. The carpets made from Control A and B are each beek dyed to a green shade of color matching as nearly as possible the color of the carpet made from the pigmented yarn (invention).

45 A sample of each carpet is tested for the resistance of its pile fibers to staining by the following 8-Hour Red Dye No.40 Staining Test:

50 An aqueous solution of cherry Kool Aid is prepared by mixing the contents of a store-bought package of cherry Kool Aid premix and water according to the instructions written on the package. The solution contains FD&C Red Dye No. 40 and citric acid. The concentration of the dye is 0.08 grams/liter and that of the acid was 0.4 grams/liter. The solution has an optical density of 3.5 and a pH of 3.0. A plastic pipe measuring about 3.8 cm in diameter and 5.0 cm in length is placed upright on and at the center of the carpet sample. Into the open end of the pipe is poured 20 ml of the prepared dye solution. The solution is left in the pipe for a period of 8 hours thereby permitting the solution to thoroughly soak into the carpet. The pipe is then removed leaving a wet red spot on the carpet sample. The spot is washed with water by placing the sample under a running tap water faucet to remove all or as much as possible of the red dye from the carpet fibers. Each spot is then graded on a scale of 1 to 8 with 1 being severely stained by the red dye and 8 being no visible stain on the carpet.

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TABLE

Carpet Fibers	8 Hr Test Rating
Invention	8.0
Control A	1.0
Control B	8.0

The results show that nylon fibers of the present invention have Red Dye No. 40 stain resistant characteristics that are equivalent to those of nylon fibers coated with stainblocker (Control B) and superior to those of the unmodified nylon fibers (Control A).

In a separate test, a sample of each carpet is exposed to store bought Clorox bleach for a period of 18 hours. The fibers of the carpet constructed from fiber of the present invention shows only minimal change in color, whereas the fibers of each of the carpets constructed from the Control fibers is bleached nearly white.

For further purposes of comparison, a yarn corresponding to Control A is prepared except that in this instance 1% by weight of the above green pigment composition is dispersed therein. The yarn is then used to make carpet in the manner described above. When a sample of this carpet is subjected to the 8-Hour Red Dye No. 40 Staining Test, the carpet is noticeably stained.

In related tests a carpet made from fibers of the present invention is subjected to the common household substances listed in the following table. The carpet is made using the same procedure described above except in this instance the phthalocyanine green pigment of the pigment/nylon 6 composition is replaced with Ferric oxide. Each substance is applied to the carpet sample, rubbed into the carpet, left on the carpet overnight and, then, the next day the carpet sample is first washed with a dilute solution of commercial detergent and then with water. All of the substances listed in the table are removed from the carpet staple.

TABLE

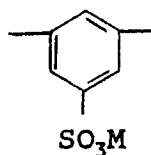
catsup	coffee	water color
chocolate	tea	grape juice
nail polish	red wine	acne medication

Claims

1. A carpet fiber comprising nylon polymer containing a pigment dispersed therein, said polymer being modified to contain aromatic sulfonated units as an integral part of its polymer chain, wherein the amount of said units is selected to provide fibers which, when subjected to the 8-Hour Red Dye No. 40 Staining Test, show no visible staining.

2. The fiber of claim 1 wherein said nylon polymer is nylon 66 polymer.

3. The fiber of claim 1 wherein said aromatic sulfonated units are of the formula



where M is an alkali metal.

4. The fibers of claim 3 wherein M is sodium or potassium.

5. The fibers of claim 1 in the form of staple.

6. The fibers of claim 1 in the form of filaments.
7. A carpet having a pile comprising the fibers of claim 1.
8. The carpet of claim 7 wherein said nylon polymer is nylon 66 polymer.

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