

1

3,630,990

TEXTILE FIBERS

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ABSTRACT OF THE DISCLOSURE

Disclosed is a textile fiber comprising poly(1,4-cyclohexylene dimethylene terephthalate) having dispersed therein a specific type of precipitated barium sulfate, the barium sulfate having an average particle size of about 0.8 micron. The fibers disclosed are characterized by improved dyeability with disperse dyes and soil-hiding properties. Also disclosed are yarns and fabrics containing the novel fibers of this invention.

This invention relates to textile fibers and yarns made therefrom which have an increased affinity for disperse dyes, improved luster characteristics and improved soil-hiding characteristics when utilized in carpets and wall coverings. More particularly, this invention relates to novel poly(1,4-cyclohexylene dimethylene terephthalate) textile fibers and yarns made therefrom, the fibers having special precipitated barium sulfate particles dispersed therein to give the above mentioned surprising improvements in the fiber.

It is known in the prior art that certain particles can be dispersed in polyester melts and fibers spun therefrom will have certain characteristics. For example, TiO_2 has been widely used as an additive to deluster man-made fibers. In particular TiO_2 has been used in poly(ethylene terephthalate) textile fibers and in poly(1,4-cyclohexylene dimethylene terephthalate) textile fibers as a delusterant. However, it is well known TiO_2 gives a chalky delustering effect to poly(1,4-cyclohexylene dimethylene terephthalate) when levels as high as about $\frac{1}{2}\%$ TiO_2 are used.

U.S. Pat. 2,924,503 discloses the use of alkaline earth sulfate filler in poly(ethylene terephthalate) to increase the rate of drawing. Further, this patent teaches that barium sulfate (type, grade, etc., unknown) added to poly(ethylene terephthalate) fibers will result in the yarns produced not being delustered.

U.S. Pat. 3,221,226 discloses that wrinkle free polyester film may be prepared by the addition of finely divided inorganic pigment to the film. Also disclosed is poly(1,4-cyclohexylene dimethylene terephthalate) having finely divided silica gel dispersed therein and spun into fibers. The fibers after being made into a yarn are reported to produce a fabric having wrinkle resistant properties. Also mentioned as useful to impart wrinkle resistance to films and fabrics are TiO_2 , BaSO_4 , talc, SiO_2 , gypsum, bentonite, Al_2O_3 , and others.

I have found that textile fibers made from poly(1,4-cyclohexylene dimethylene terephthalate) having a special precipitated barium sulfate therein (to be described in detail later) unexpectedly react differently as to dyeability and soil hiding than does poly(ethylene terephthalate) having the same additive. Further, I have found that TiO_2 or silica gel when added to poly(1,4-cyclohexylene dimethylene terephthalate) do not give the improved dyeability and soil hiding properties to poly(1,4-cyclohexylene dimethylene terephthalate) that are obtained from the use of the special precipitated barium sulfate in poly(1,4-cyclohexylene dimethylene terephthalate). This aspect of the invention will be illustrated by specific examples in greater detail hereinafter.

A major disadvantage which is associated with the use

2

of man-made fibers in carpets is that the optical properties of the fibers tend to show soil on and beneath the surface of the carpet. The fibers act as a lens and magnify the dirt or soil particles lying on the opposite side of the fiber. Many attempts have been made to change these properties and still retain luster and performance characteristics of the fiber. Novel cross-section fibers have been used and are still in general use for this purpose. A drawback of the novel cross-section approach with prior art fibers is that the shallow channels found along the surface of the fibers tend to wick soiling materials such as liquids along the fiber to make the small spots spread into larger spots.

The novel fibers of this invention can be made having a conventional round cross-section and still obtain excellent soil-hiding properties without the problem of wicking of liquids spilled on carpets made from the fiber. Of course, the novel fibers of this invention can be made in any cross section desired to obtain both the advantages of my novel fiber and the advantages of the novel cross section fibers if desired.

A second problem which carpet manufacturers must deal with is the dyeability of man-made fiber. In this respect it is the usual procedure in open beck dyeing poly(1,4-cyclohexylene dimethylene terephthalate) fiber with disperse dyes to use a carrier such as a biphenyl carrier in the amount of about 10% by weight. The novel fibers of this invention can be dyed with disperse dyes using a lower level of carrier and thus effect a cost savings for the carpet manufacturer.

Another problem which has plagued carpet manufacturers since the advent of synthetic resinous polymer fibers has been one of trying to obtain the proper luster for the fiber without adversely affecting the physical properties thereof. The most commonly used delusterant is TiO_2 which has been used in poly(ethylene terephthalate) and poly(1,4-cyclohexylene dimethylene terephthalate) fibers. However, to obtain proper luster the carpet manufacturers have traditionally found it necessary to blend bright fiber with semi-dull fiber to overcome to a degree the chalky appearance of a semi-dull fiber which has TiO_2 as a delusterant. I have found that by using a particular barium sulfate in poly(1,4-cyclohexylene dimethylene terephthalate) that I avoid the chalky appearance before and after dyeing and thus can provide a single luster fiber and eliminate the need for a blending step in making spun carpet yarn from poly(1,4-cyclohexylene dimethylene terephthalate) fiber.

It is an object of this invention to provide novel poly(1,4-cyclohexylene dimethylene terephthalate) textile fibers and yarns which exhibit an improved affinity for dyes and which have improved soil hiding characteristics as compared to conventional poly(1,4-cyclohexylene dimethylene terephthalate) textile fibers.

My novel poly(1,4-cyclohexylene dimethylene terephthalate) fibers are obtained by adding to the poly(1,4-cyclohexylene dimethylene terephthalate) polymer, by any convenient means prior to final extrusion, at about 0.3% or more, by weight, of special precipitated barium sulfate, the fibers then being extruded and subsequently processed in the conventional manner to produce staple fibers or continuous filaments suitable for use in carpets.

A major advantage of the novel fibers of this invention is improved soil-hiding properties without sacrifice of performance characteristics. Another advantage is the enhanced dye-ability of the fibers with disperse dyes thus resulting in substantial savings in dyeing costs.

When referring to barium sulfate hereinafter, it is to be understood that I am referring to a special precipitated barium sulfate known as blanc fixe barium sulfate described as follows. In the trade the term blanc fixe is reserved for precipitated barium sulfate as opposed to

ground baryte type barium sulfate. The term "baryte" is used to identify the natural form or mineral barium sulfate. It is practically impossible to reach the state of subdivision which characterizes blanc fixe barium sulfate by any known method or grinding barytes.

The precipitation process used to make blanc fixe barium sulfate particles uses a solution in the precipitation which has been purified before precipitation. The

EXAMPLE 1

Poly(1,4-cyclohexylene dimethylene terephthalate) and poly(ethylene terephthalate) fibers were melt-spun, drawn in a heated environment, crimped, heat-set with free fiber shrinkage, lubricated, and cut into 6-inch staple in a manner well known to those skilled in the art. A description of each fiber lot is as follows:

Sample number	W-2772	W-2771	W-2770	W-2642	W-2641
Polymer.....	CHDMT	CHDMT	CHDMT	PET	PET
Percent BaSO ₄ (by weight) ¹	None	2.75	2.75	None	2.75
Percent OB-1 (by weight) ¹	do.	0.02	None	do.	None
Fiber cross-section.....	Round	Round	Round	Round	Round
Fiber IV.....	0.68	0.68	0.64	0.59	0.56
Draw ratio ²	3.4	3.4	3.4	5.7	5.7
Heat-setting temperature (° C.) ²	215	215	215	145	145
Drawn, denier/filament.....	15	15	15	15	15
Filament strength (grams/denier) ³	2.39	2.32	1.78	4.77	4.15
Percent elongation ³	33.7	34.9	24.8	38.5	35.0

¹ Added during polymerization. The barium sulfate with about 0.8 micron average particle size was used. The barium sulfate can be incorporated prior to final extrusion into filaments in a variety of ways, such as a glycol slurry, dry mixing, master batch, etc.

² Draw ratio and heat-setting temperature were selected to produce optimum properties for carpet performance.

³ Tested on an Instron with one-inch gauge length, one inch per minute crosshead speed, ten-inches per minute chart speed, and seventy-five grams full scale load.

purification controls the purity of the product to give excellent whiteness which characterizes blanc fixe barium sulfate. The conditions of precipitation such as, concentration, temperature, pH, method of combining solutions and after-treatments such as washing, filtering, drying and grinding also play a necessary role in controlling whiteness.

The fibers were then spun on the worsted system into 1.1/1 cotton count, 2.75 S twist carpet yarns and were tufted into 34 oz./sq.yd., loop-pile carpets with a 3/8" pile height. The cones of yarn were arranged in the tufting machine creel so as to be tufted in bands simultaneously as shown in the following outline:

Carpet identification No.	Band 1	Band 2	Band 3	Band 4
CT 2899-25.....	CHDMT	CHDMT	PET	PET
(Carpet No. 1).....	Bright (control)	2.75% BaSO ₄	2.75% BaSO ₄	Bright (control)
CT 2899-26.....	CHDMT	CHDMT	PET	PET
(Carpet No. 2).....	Bright (control)	2.75% BaSO ₄ , 0.02% OB-1	2.75% BaSO ₄	Bright (control)
CT 2899-27.....	CHDMT	CHDMT	CHDMT	CHDMT
(Carpet No. 3).....	Bright (control)	2.75% BaSO ₄ , 0.02% OB-1	2.75% BaSO ₄	Bright (control)

The particle shape of the blanc fixe barium sulfate crystal is at least one of six recognizable distinct shapes. The distinct shapes are: spindle, diamond, sphere, rectangular, bar, and star shapes. Mosaic structures occur by small particles dissolving and crystallizing again on larger particles and cementing them together. Microscopic examination and electron photomicrographs of the blanc fixe barium sulfate show the rectangle and diamond but the mosaic structure dominates. This conclusion is also borne out by the PSD (particle size distribution) which prove it to have an average size of just under one micron while the measured size taken from electron photomicrographs show the individual crystals to have an average size of .13 to .18 of a micron.

A representative particle size distribution of the type of blanc fixe barium sulfate is as follows:

	Percent
Smaller than 0.5 micron	18
0.5-1 micron	60
1-1.5 microns	14
1.5-2 microns	6
2-2.5 microns	1
2.5-5.5 microns	0.5
Greater than 5.5 microns	0.5
	100.0

When OB-1 is referred to in the example I am referring to an optical brightener of the type disclosed in U.S. Pat. No. 3,260,715. The particular optical brightener I have used is the fluorescent compound 4,4'-bis(benzoxazol-2-yl) stilbene.

My invention is illustrated by the following examples of preferred embodiments thereof. The advantages are illustrated by comparative examples which illustrate the differences between my invention and the prior art known in the field of this invention.

Carpet No. 1 was piece-dyed honey beige using the following polyester dyeing procedure:

(A) Dye bath components by weight on total carpet weight:

	Percent
Eastman Polyester Brilliant Red FFBL-Disp. Red	
60	0.048
Eastman Polyester Yellow 2R-Disp. Yellow	
86	0.35
Eastman Polyester Blue BLF-Disp. Blue 120	0.44
Carolid 3F (biphenyl carrier)	10
Monosodium phosphate	1
Calgon (sodium hexamethaphosphate)	1

(B) Hot rinse prior to dyeing in water at 160° F. for 10 minutes.

(C) Fill dye beck with water (30 to 1 ratio of water to carpet by weight). Carpet remains in the beck.

(D) Raise water temperature to 110° F. and add dye bath components except Carolid 3F biphenyl carrier.

(E) Raise temperature (5°/minute rate of rise) to 160° F. and add Carolid 3F biphenyl carrier.

(F) Raise temperature (5°/minute rate of rise) to the boil and dye for one hour.

(G) Cool to 160° F. and drain bath.

(H) Refill with water and rinse at 160° F. for 10 minutes.

(I) Rinse with softener at 120° F. for 20 minutes [2% Ceranue HC (cationic lubricant) concentrate plus 1% acetic acid].

(J) Drain, remove carpet, extract and dry carpet at 250° F.

(K) Apply latex and complete the "double-backing" process as explained later herein.

The four bands in Carpet No. 1 were graded visually (5 judges) and were tested for color difference (E) using an I.D.L. "Color Eye" machine. The results showed that

5

poly(1,4-cyclohexylene dimethylene terephthalate) sample containing 2.75% barium sulfate dyed a significantly deeper, richer shade with a pleasing, subdued luster compared to the poly(1,4-cyclohexylene dimethylene terephthalate) fiber without barium sulfate. The poly(1,4-cyclohexylene dimethylene terephthalate) sample without barium sulfate dyed a deeper richer shade than the poly(ethylene terephthalate) samples with and without barium sulfate. The poly(ethylene terephthalate) fiber sample containing barium sulfate was clearly off-shade, dyeing a light, chalky-gray beige compared to the other samples.

EXAMPLE 2

The three carpets described in Example 1 were mock-dyed in order to maintain the natural luster of the fibers for a soiling test. In mock-dyeing, the normal dyeing procedure was used as described previously with the exception that the dyestuff was not included. Latex was then applied to the jute primary backing in the normal manner to hold the tufts in place. The samples were given a standard double-backing to produce the desired dimensional stability. "Double-backing" is a term used to describe the well-known process of pressing a second piece of jute (secondary backing) against the freshly-latexed primary jute and drying at about 250° F. to 310° F. to lock the tufts and bond the two pieces of jute together. Materials other than jute are commercially available for use as primary and secondary backing.

In order to determine the soil-hiding performance of my novel fibers compared to the poly(ethylene terephthalate) fibers, with and without barium sulfate, and the bright poly (1,4-cyclohexylene dimethylene terephthalate), the mock-dyed carpets identified as CT 2899-25, CT 2899-26, and CT 2899-27 were placed on the floor to be exposed to office traffic for 5000 steps (recorded on a counter automatically). The carpets were rotated each day and were cleaned once each day with a Hoover Dial-A-Matic vacuum cleaner. Whiteness and whiteness difference values were obtained on the original carpets and after floor testing using an I.D.L. "Color Eye" machine (manufactured by the Instrument Development Laboratories, a division of Kollmorgen Corp. of Attleboro, Mass.). Results were as follows, arranged in order of decreasing whiteness value:

Sample No.	Description of fiber	Original	After floor test
W-2771.....	CHDMT plus 2.75% BaSO ₄ plus 0.02% OB-1.	112.6	45.0
W-2770.....	CHDMT plus 2.75% BaSO ₄	64.7	25.2
W-2641.....	PET plus 2.75% BaSO ₄	62.1	20.2
W-2772.....	CHDMT bright.....	47.4	16.0
W-2642.....	PET, bright.....	42.1	16.9

NOTE.—A difference of 5% is readily detectable visually and is significant statistically.

These soiled samples clearly demonstrate the superiority in soil-hiding power and overall appearance of Sample No. W-2771 and Sample No. W-2770 over the other samples.

Similar comparative results were obtained in an 18,000 step floor test. In the 18,000 step test, it became apparent that Samples W-2770, W-2771 and W-2772 had superior texture retention compared to W-2641 and W-2642. Sample No. W-2642 had a smeared appearance after floor testing. The cleanability and texture restoration of W-2770 and W-2771 were excellent.

The most important point as clearly shown in the 18,000 step test is that my novel fiber produces excellent soil-hiding, cleanability and texture restoration maintaining excellent tuft identity while the poly(ethylene terephthalate) fiber containing barium sulfate particularly after about 18,000 steps has an undesirable smeared appearance and loss of tuft identity after soiling which could not be satisfactorily corrected by vacuuming and cleaning.

EXAMPLE 3

The four bands in Carpet No. 1 (CT 2899-25) were tested for relative printability by padding on a blue print

6

paste (70% wet pick-up), steaming eight minutes at 212° F. to fix the dyestuff and scouring to remove excess paste (20 min. at 180° F.). Poly(1,4-cyclohexylene dimethylene terephthalate) with and without barium sulfate produced essentially the same results in terms of depth of shade. The poly(ethylene terephthalate) bright fiber produced a slightly darker shade than both poly(1,4-cyclohexylene dimethylene terephthalate) fibers (with and without barium sulfate). However, the poly(ethylene terephthalate) with barium sulfate dyed significantly lighter than the other three fibers. All four yarns were tufted side-by-side in bands in the same carpet and were padded, steamed and scoured simultaneously to insure identical treatment. The results clearly show that barium sulfate does not adversely affect printability of poly(1,4-cyclohexylene dimethylene terephthalate) while causing a significant loss in depth of shade in the poly(ethylene terephthalate) fiber.

The formulation used in the printing test was as follows:

(A) Dyestuff (print paste):

	Grams/liter
Eastman Polyester Blue BLF-Disp. Blue 120 ¹	4
Dowanol EPH-ethylene glycol phenyl ether	50
Cham Carrier L-1300-xylene-biphenyl mixture	50
Progowet R-60-diocylsulfosuccinate	50
Polygum 260-purified natural gum ether	6

(B) Scour:

	Grams/liter
Caustic	2
Sodium hydrosulfite	2
Nonionic detergent	2

¹ Grams/liter of solution.

EXAMPLE 4

In order to remove any differences in dyeing related to polymer type, a carpet was prepared containing a band of yarn composed of poly(ethylene terephthalate) bright fiber and a band of yarn containing poly(ethylene terephthalate)+2.75% barium sulfate. A separate carpet was prepared containing a band of poly(1,4-cyclohexylene dimethylene terephthalate) bright fiber and a band of poly(1,4-cyclohexylene dimethylene terephthalate)+2.75% barium sulfate. Each of these two carpets was dyed separately a honey beige shade using the dyeing procedure previously described with the following dyestuff:

	Percent
Eastman Polyester Red FFBL-Disp. Red 60	0.048
Eastman Polyester Yellow 2R-Disp. Yell. 86	0.35
Eastman Polyester Blue BLF-Disp. Blue 120	0.44

Thus poly(ethylene terephthalate) with and without barium sulfate was dyed in one bath and poly(1,4-cyclohexylene dimethylene terephthalate) with and without barium sulfate was dyed in another bath. The carpets were graded for comparative dyeing performance with the following results:

The poly(ethylene terephthalate) sample with barium sulfate was clearly off-shade. Both were lighter than the poly(1,4-cyclohexylene dimethylene terephthalate) samples. Poly(1,4-cyclohexylene dimethylene terephthalate) with barium sulfate dyed a significantly deeper, richer shade with a pleasing, subdued luster compared to the poly(1,4-cyclohexylene dimethylene terephthalate) bright carpet. Thus it was concluded that barium sulfate does not have the same effect on the dyeing of poly(ethylene terephthalate) as it does on poly(1,4-cyclohexylene dimethylene terephthalate). Honey beige is a typical carpet color. Thus my work to date in dyeing and printing has shown that barium sulfate has a beneficial effect on poly(1,4-cyclohexylene dimethylene terephthalate) but an adverse effect on poly(ethylene terephthalate).

EXAMPLE 5

It is well established in the trade that 10% biphenyl carrier (based on total weight of carpet yarn and primary

backing) is needed in open-beck dyeing to achieve the desired depth of shade and level piece-dyeing with prior-art poly(1,4-cyclohexylene dimethylene terephthalate) and poly(ethylene terephthalate) fibers in bright lusters as well as semi-bright and semi-dull lusters produced by incorporating TiO_2 in the fibers. I have found that my novel poly(1,4-cyclohexylene dimethylene terephthalate) fiber containing barium sulfate can be dyed with less carrier than the poly(1,4-cyclohexylene dimethylene terephthalate) and poly(ethylene terephthalate) fibers of prior art.

The previously described procedure was used to piece-dye a poly(ethylene terephthalate) carpet and a poly(1,4-cyclohexylene dimethylene terephthalate) carpet honey beige using only 4% carrier instead of 10%. Honey beige is in the light-medium shade range. The poly(ethylene terephthalate) fiber carpet had a band of poly(ethylene terephthalate) fibers and a band of poly(ethylene terephthalate) fibers having 2.75% barium sulfate therein. The poly(1,4-cyclohexylene dimethylene terephthalate) fiber carpet had a band of poly(1,4-cyclohexylene dimethylene terephthalate) fibers and a band of poly(1,4-cyclohexylene dimethylene terephthalate) fibers having 2.75% barium sulfate therein.

The poly(ethylene terephthalate) carpet was dyed separately from the poly(1,4-cyclohexylene dimethylene terephthalate) carpet. Dyeing results were as follows:

(A) Poly(ethylene terephthalate)—Slightly darker than poly(ethylene terephthalate) with barium sulfate. Dyeing was off-color, uneven and mottled. Unsatisfactory.

(B) Poly(ethylene terephthalate)+2.75% barium sulfate—Dyed lightest. Dyeing was off-color, uneven and mottled. Unsatisfactory.

(C) Poly(1,4-cyclohexylene dimethylene terephthalate)—Lighter than poly(1,4-cyclohexylene dimethylene terephthalate) with barium sulfate.

(D) Poly(1,4-cyclohexylene dimethylene terephthalate)+2.75% barium sulfate—Dyed darkest. A rich honey beige color was obtained.

EXAMPLE 3

Four bands of Carpet No. 1 (CT 2899-25) and Band 2 of Carpet No. 2 (CT 2899-26) were cut to form five separate samples. These five carpets were dyed separately for 90 minutes at 190° F. using 8.5% carrier and the following dark red dye formulation:

	Percent
Eastman Polyester Dark Red FL-Disp. Red 65	0.90
Eastman Polyester Red FFBL-Disp. Red 60	0.60

The detailed procedure previously described was used and additionally the following scour was used prior to the rinse:

	Percent
Caustic (soda ash)	2
Emulphor EL 719-(polyoxyethylated vegetable oil)	2

The carpet was scoured for twenty minutes prior to the rinse. Results were as follows:

(A) Poly(ethylene terephthalate)—Dyeing was light and off-shade. Slightly darker than poly(ethylene terephthalate) with barium sulfate.

(B) Poly(ethylene terephthalate)+2.75% barium sulfate—Dyed lightest. Dyeing was light and off-shade.

(C) Poly(1,4-cyclohexylene dimethylene terephthalate)—Dyed lighter than poly(1,4-cyclohexylene dimethylene terephthalate) with barium sulfate (with and without OB-1) but darker than poly(ethylene terephthalate) and poly(ethylene terephthalate) with barium sulfate. Color was slightly off-shade, not as rich as the two carpets of poly(1,4-cyclohexylene dimethylene terephthalate) with barium sulfate.

(D) Poly(1,4-cyclohexylene dimethylene terephthalate)+2.75% barium sulfate+0.02% OB-1 and poly(1,4-cyclohexylene dimethylene terephthalate)+2.75% barium

sulfate—Dyed darkest. A rich, deep, red color was obtained.

Similar comparative results were obtained when the test was repeated with 0.75% Standpon alkylaryl polyglycol ether added as a leveling agent. The dyeing differences persisted even when the bath temperature was raised to the boil.

The results of these tests show the superiority of poly(1,4-cyclohexylene dimethylene terephthalate) with barium sulfate over poly(ethylene terephthalate) with and without barium sulfate as well as poly(1,4-cyclohexylene dimethylene terephthalate) fibers of prior art. Poly(1,4-cyclohexylene dimethylene terephthalate) with barium sulfate has superior dyeing characteristics not only at the 10% carrier level and 205° F. to 212° F. dyeing temperature but also my novel fiber can be dyed at reduced carrier levels and lower temperatures. In some shades, generally light to medium, less than 5% carrier can be used. In other shades, my novel fiber can be dyed with less than 10% carrier. The cost reduction in dyeing obtained by using poly(1,4-cyclohexylene dimethylene terephthalate) containing barium sulfate is obvious. Procedure and formulation vary somewhat from one dye house to another, but a reduction in the amount of a costly component required, such as carrier, offers a significant economic advantage.

EXAMPLE 7

In a comparative carpet performance test between poly(1,4-cyclohexylene dimethylene terephthalate) fibers with and without barium sulfate and poly(ethylene terephthalate) fibers with and without barium sulfate, the following results were obtained using $\frac{3}{8}$ inch pile, level loop carpets containing 34 oz./sq. yd. of 1.1/1 cotton count, 2.75 S twist yarns tufted into jute (9 oz./sq. yd.). The carpets were mock-dyed, double-backed with jute as previously described and tested. Results of this test were as follows using a 1 to 5 rating system with 1 being poor and 5 excellent:

Test	Carpet description			
	PET, bright	PET plus 2.75% BaSO ₄	CHDMT, bright	CHDMT plus 2.75% BaSO ₄
Texture retention after 20,000 steps	3	2	5	5
Static load recovery after 24 hours (100 p.s.i. for 1 week)	2	2	5	5

This example illustrates that the novel fibers of my invention while gaining dyeability with disperse dyes and gaining soil-hiding characteristics do not lose desirable characteristics necessary for high performance in carpets. Note however, that barium sulfate in poly(ethylene terephthalate) fiber does have an adverse effect on carpet performance characteristics tending to produce the aforementioned smeared appearance.

EXAMPLE 8

The following samples were melt-spun and processed into oriented, crimped, and heat-set staple fibers:

Polymer	Cross-section	Percent BaSO ₄	Percent TiO ₂
CHDMT	Round	3.0	
CHDMT	Novel	3.0	
CHDMT	Round		0.1
CHDMT	Novel		0.1
PET	Round	3.0	
PET	Round		0.1

The poly(1,4-cyclohexylene dimethylene terephthalate) and poly(ethylene terephthalate) were processed as previously discussed in Example 1. Worst-spun 2.25/2 yarns were prepared from each fiber lot. The yarns were tufted into 40 oz./sq. yd., $\frac{1}{4}$ inch pile, $\frac{1}{8}$ inch gauge commercial-grade carpets. One-third of each tufted sample was

mock-dyed (that is, all dyeing procedure was used except no dyestuff was added) to leave the carpets in the finished state but in natural color. The remainder of each tufted sample was divided into two sections. One section was dyed dark blue (9% carrier, 205° F., 90 minutes) and the other dark red (10% carrier, 205° F., 90 minutes). Each of the carpets was dyed separately, scoured as described in Example 6 and finished in a final rinse containing 2% Ceranine HC softener (cationic lubricant) and 1% acetic acid (percent based on total weight of carpet). The carpets were rinsed for 20 minutes at 120° F., dried, latexed, and double-backed with jute in a manner well known in the carpet trade.

The mock-dyed carpets were floor-tested to 50,000 steps with gradings for soiling at 5,000; 10,000; 20,000 and 50,000 measured steps. Samples were ranked for soiling using a 1 to 5 point grading system with 1 being the worst (poor) and 5 being the best (excellent). A similar grading system was used for texture retention. The following floor-test results were obtained:

Sample	Polymer	Cross section	Percent BaSO ₄	Percent TiO ₂	Texture retention	Clean ability and texture restoration
B	CHDMT	Non-round	3		4	5
A	CHDMT	Round	3		5	5
E	PET	do.	3		2	3
D	CHDMT	Non-round		0.1	4	2
C	CHDMT	Round		0.1	5	2
F	PET	do.		0.1	2	1

It was observed in grading the dyed carpets that poly(ethylene terephthalate) Samples E and F were off-shade due to relatively light dyeing. Sample E was found to be more off-shade than Sample F. Poly(1,4-cyclohexylene dimethylene terephthalate) Samples B and A dyed rich, deep colors with B having a relatively sparkling (light-reflective) luster and Sample A having a pleasing subdued luster. Samples C and D dyed lighter than A and B, respectively, but darker than Samples E and F.

This test demonstrated the superiority in cleanability, texture restoration and dyeability of poly(1,4-cyclohexylene dimethylene terephthalate) containing barium sulfate in round and non-round (such as trilobal or trilobal) cross-sections compared to the fibers of prior art. In addition, the texture retention of my novel fibers after floor-testing was found to be very good (4) to excellent (5). Not all cross-sections produced these outstanding overall results, however. My novel fiber in 2/1 "H" hollow and roughly equilateral multi-lobal cross-sections produced satisfactory overall results in a variety of carpet styles. In certain other cross-sections (3/1 "Y," 4/1 "Y," flat ribbon and 3/1 "L"), the fibers were more difficult to crimp and spin into uniform yarns and, in addition, carpets made from these fibers produced more crushing and poorer texture retention in loop-pile carpets.

EXAMPLE 9

Surprisingly, it has been found that poly(1,4-cyclohexylene dimethylene terephthalate) containing as little as about 0.3% barium sulfate with or without an optical brightener (OB-1) has superior dyeing characteristics compared to the prior art fibers. For this test, the following 1.1/1, 3.0 S worsted-spun yarns were tufted into 32 oz./sq. yd., 3/8 inch, loop-pile carpets:

Sample	Polymer	Cross section	Percent TiO ₂	Percent BaSO ₄
A	CHDMT	Round		0.3
B	CHDMT	Non-round		0.3
C	CHDMT	Round		10.3
D	CHDMT	do.	0.1	
E	PET	do.		0.3
F	PET	do.	0.1	

¹ Plus 0.01% OB-1.

The carpets were dyed in the same manner as described in Examples 5, 6 and 8 with remarkably similar results

for such large differences in amounts of barium sulfate. A semi-bright luster was obtained in Samples A, D, E, and F. Sample B had a relatively sparkling (light reflective) luster and Sample C had a brilliant white natural luster.

It was found that unusual differential luster effects can be obtained by producing a plied yarn containing at least one ply spun from Sample B and at least one ply of yarn spun from Samples A or C and thereafter tufting, weaving, or otherwise processing the yarn to form a carpet or wall covering.

An intimate blend of Samples A and B (or other combinations) can be obtained by extruding at least a portion of Sample B from part of at least one multi-hole spinneret and at least a portion of Sample A from the same spinneret and further processing to form staple fibers and bulked continuous filament yarns as desired. Another acceptable procedure is to combine the output of several spinnerets each of which is designed to extrude either Sample A or Sample B in any desired proportions and subsequently to orient, crimp, and further process

Samples A and B together to form staple or bulked continuous filament yarn as desired. These same processes for producing fiber differential luster effects and intimate blends can be applied to poly(1,4-cyclohexylene dimethylene terephthalate) containing higher levels of barium sulfate with or without an optical brightener.

My aforementioned novel fibers can be used in a wide variety of commercial and domestic carpet styles and wallcoverings. These carpets and wallcoverings can be produced by tufting, weaving, knitting, needle-punching and the other processes. Also at low levels of barium sulfate additive the fibers can be produced in fine denier for other end uses where enhanced disperse dyeability and luster characteristics are desired. At high levels (approximately 4-6% by weight) particularly where an optical brightener such as OB-1 is used my novel fibers can be used to produce dyed knit fabrics that have a subtle subdued luster resembling wool.

The invention has been described in detail with particular reference to preferred embodiments thereof, but it will be understood that variations and modifications can be effected within the spirit and scope of the invention as described hereinabove.

I claim:

1. Textile fiber comprising poly(1,4-cyclohexylene dimethylene terephthalate) having dispersed therein from about 0.3% to about 6.0%, by weight, of precipitated barium sulfate particles, said precipitated barium sulfate particles ranging in size up to about 2.0 microns.

2. Textile fiber of claim 1, wherein the majority of said precipitated barium sulfate particles range in size up to about 2 microns.

3. Textile fiber of claim 2 having at least 0.01% by weight of the fluorescent compound 4,4'-bis(benzoxazol-2-yl) stilbene dispersed therein.

4. Textile fiber of claim 1, wherein the precipitated barium sulfate particles have an average size of about 0.8 micron.

5. Textile fiber of claim 1, wherein said barium sulfate particles are present in the amount of at least 2.75% by weight.

6. Textile fiber of claim 4, wherein the denier per filament of said fiber is from about 10 to about 20.

11

- 7. Textile fiber of claim 4 having at least 0.01% by weight of the fluorescent compound 4,4'-bis(benzoxazol-2-yl) stilbene dispersed therein.
- 8. Textile fiber of claim 5, wherein the average particle size of said barium sulfate is about 0.8 micron.
- 9. Textile fabric containing fiber of claim 2.
- 10. Textile fabric containing fiber of claim 3.
- 11. Textile fabric containing fiber of claim 6.
- 12. Textile yarn comprising spun staple fiber made from poly(1,4-cyclohexylene dimethylene terephthalate) having dispersed therein from about 3.0% to about 6.0%, by weight, of precipitated barium sulfate having an average particle size of about 0.8 micron and ranging in size up to about 2.0 microns.
- 13. Textile yarn comprising continuous filaments of poly(1,4 - cyclohexylene dimethylene terephthalate) having dispersed therein from about 3.0% to about 6.0%,

12

- by weight, of precipitated barium sulfate having an average particle size of about 0.8 micron and ranging in size up to about 2.0 microns.
- 14. Textile fiber of claim 4, wherein said fiber is a carpet fiber of about 15 denier per filament.

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