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POLYPROPYLENE CARPET FIBERS

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This invention relates to textile carpets made with polypropylene fibers. In another aspect it relates to providing a carpet of superior tuft resilience woven from polypropylene fiber.

Thermoplastics have been considered, and certain types developed, as a source of textile fibers, but they have also been found to present problems which were long ago solved with the older cellulosic fibers. Polyolefins, among other thermoplastics, because of their chemical resistivity are a class of textile yarns of highly desirable properties, if the physical processing requirements can be attained. Polyolefins can be readily prepared as a continuous single filament by extrusion from a polymer melt through a die. It is then possible to form a multifilament yarn therefrom in many textile applications.

One of the main drawbacks with natural fibers and conventionally prepared polyolefin fibers has been their lack of long-term resilience when employed in carpets. Typically, a polyolefin staple yarn, or bulked continuous filament yarn, when manufactured by conventional methods, tends to pack down after as little as several days of depression under furniture legs, and the like, retaining an unsightly permanent set when the furniture is moved. It is hypothesized that this may occur because, under a long-term load, the polyolefin molecules and/or crystallites tend to slip past one another leading to a more or less permanent deformation.

The prior art has suggested the use of ionizing radiation, such as high energy electrons, to cause cross-linking in formed polyethylene and give a less deformable plastic shape. It should be noted, however, that the use of ionizing radiation is relatively expensive, difficult to control, and being hazardous, requires considerable safeguards.

The cross-linking of polyethylene with radiation has been extensively studied; it is known to be a polymer of the cross-linking type with a scission-to-crosslinking ratio of about 0.35. Radiation affects polypropylene quite differently than it does polyethylene. Whereas the crosslinking-to-scission ratio is favorable for crosslinking of polyethylene, it is very close to unity for polypropylene, and yields a very low efficiency for the crosslinking of the latter under straight radiation-crosslinking. The dose required for incipient gelation (indicative of cross-linking) of polypropylene, for example, is reported to be about 50 megarads even though this dose has been decreased with annealing techniques. Keeping in mind the expense of high energy radiation, the cross-linking of formed polypropylene by this method would appear economically prohibitive.

It has been discovered that in order to economically cross-link polypropylene, it is first necessary to incorporate a cross-linking aid, specifically a polyfunctional monomer, into the polypropylene yarn, which monomer cross-links upon exposure to radiation and gives a fiber of significantly greater resilience, and thus improved resistance to permanent deformation under load than unmodified polypropylene fiber.

According to the present invention, a conventionally manufactured polypropylene continuous filament, or staple yarn is first bulked, e.g. by crimping, to give a bulked yarn, and then may be cross-linked by a method which comprises: immersing the yarn in a bath of a

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polyfunctional monomer selected from the group consisting of divinyl benzene, the mono-, di-, tri- and tetra-ethylene glycol diacrylates, the mono-, di-, tri- and tetra-ethylene glycol dimethacrylates, vinyl acrylate, vinyl methacrylate, allyl acrylate, allyl methacrylate, the divinyl ether of diethylene glycol, diallyl maleate, diallyl itaconate, diallyl malonate, diallyl benzene phosphonate, triallyl phosphate, triallyl cyanurate, glyceryl trimethacrylate; subsequently exposing the monomer-saturated yarn to a field of high energy radiation for a time sufficient to give superior resilience to said yarn by cross-linking of said monomer-polymer components. The immersion may occur after as well as before the bulking step.

Fibers, especially the monofilaments used in my invention, have a diameter of 0.1 to 50 mils and more usually about 0.5 to 3 mils. The tensile strength of these fibers cold drawn is above 30,000 p.s.i., frequently above 90,000 to 100,000 p.s.i. and can range as high as 150,000 to 250,000 p.s.i. measured at a temperature in the range of 65 to 100° F.

Practice of the present invention permits cross-linked polypropylene compositions to be obtained with the utmost expedience and economy. The supplementing cross-linking effect of the absorbed copolymerizing monomer permits a desired degree of cross-linking to be obtained in the resulting composition, with greatly reduced dependence upon the effects and required quantities of the high energy radiation.

Generally, exceptionally satisfactory results can be achieved when divinyl benzene, mono-, tri-, and tetra-ethylene glycol dimethacrylate, allyl acrylate and methacrylate, vinyl methacrylate and diallyl maleate are employed as the polyfunctional monomer to cross-link, or facilitate the crosslinking of, the polypropylene fiber that is utilized. Surprisingly enough, as is apparent from the foregoing, selection of the polyfunctional monomer is relatively critical, and surprisingly non-predictable. Random choice of many ostensibly similar or equivalent polyfunctional monomers generally is found to provide inexplicably unsatisfactory (or no) results.

The quantity of the polyfunctional monomer that is incorporated in the polymer may not only depend upon the specific nature and characteristics of the particular polypropylene that is intended to be cross-linked, as well as the particular cross-linking efficiency of the polyfunctional monomer that is involved, but also upon the properties that may be desired to be imparted in the resulting composition. The quantity of the polyfunctional monomer that is employed should be adequate to secure the desired extent of cross-linking in the polymeric composition, taking into account the degree of cross-linking that will be directly induced in the composition from its exposure to the radiant high energy which serves to cross-link the starting polymer by graft copolymerization of the absorbed monomer.

Generally, a satisfactory result may be achieved when minor proportions of the monomer are incorporated intimately in the polymer. In some instances, very minor proportions may suffice, especially when low levels of cross-linking are desired in the composition. It is difficult to generalize on the weight proportions that may be suitably utilized in all instances due, as may be readily appreciated, to the great variations that may be encountered in the equivalent weights of the many polyfunctional monomers that may be utilized.

Broadly speaking, it is desirable to utilize such a quantity of the monomer as may be adapted to provide between one and about three hundred functional, cross-linking bridges per every three thousand carbon atoms in the chains of the polymer that is desired to be cross-linked. To the attainment of such ends, it may frequently be found

advantageous to incorporate an amount of the monomer in the polymer that is between 1 and 40 percent by weight, based on the weight of the resulting composition. An amount of the monomer that is between 3 and 10 percent by weight may even be more advantageous for most cross-linking purposes.

The monomer may be intimately incorporated in the polymer in any other desired manner e.g., a vapor bath, prior to the mutual irradiation of the uniformly associated mixture. Thus, physical interblending means such as may be achieved by any of a variety of conventional techniques may be utilized. In many instances, however, it may be convenient and quite suitable to incorporate a liquid monomer having a dissolving, plasticizing, or swelling influence on the polymer, by immersing the latter in the former until a desired monomer content has been uniformly attained.

The radiation which is employed for cross-linking the treated yarn is preferably of the high energy ionizing radiation, such as provided by gamma rays, electrons, protons, and alpha particles. The preferred form of radiation is gamma rays. When gamma rays are employed, the dose rate can be within the range between 1×10^3 and 1×10^{12} rads (the amount of radiation per hour producing an absorption of 100 ergs/gram) for a sufficient length of time so that the total dosage received by the fiber is within the range between 0.05 megarad up to 50 megarads, preferably in the range one to ten megarads. The total dosage necessary to form a fiber of superior resilience is primarily a function of radiation sensitivity of the particular cross-linking aid undergoing irradiation, and can be determined empirically.

Any suitable source of gamma rays can be used, including such sources as spent fuel elements from nuclear reactors, radioactive isotopes, cathode tubes, and linear accelerators employing targets such as tungsten for the conversion of electrons to gamma rays.

If electrons, protons, or alpha particles are employed, the polymer is subjected to these particles having energies of from 1 million electron volts (mev.) to 1 billion electron volts (bev.) and can be supplied from such sources as linear accelerators, Van de Graff generators, betatrons, cyclotrons, and the like. Beam currents from these sources will range from 0.1 to 1000 microamperes, and the irradiation time using such beams will vary from 0.5 to 60 minutes.

Textile crimping involves continuously pressing the filament or tow through a stuffing box, thereby compressing the material and giving creases thereto, which give bulk to the thus treated material. Other conventional bulking techniques, coiling, curling and looping, may readily be employed.

The fibers can, of course, be dyed to any of a variety of colors by method recently disclosed in the art.

The polypropylene yarns treated according to the method of this invention are now suitable for weaving into fine carpets on modern power looms. One of the principal varieties of carpets that can be manufactured with polypropylene yarn is domestic Axminster. A firm groundwork of jute or cotton is employed, upon which the pile containing the design is fastened in tufts of soft polypropylene yarn. These tufts are supplied from a series of rollers corresponding in number to the picks or wefts completing a pattern, and in length to width of the carpet, and are independent of the warp and woof comprising the body of the fabric. This permits the employment of an almost unlimited number of colors and the most perfect and elegant designs.

Wilton carpets, another major type, are woven alike, and of the same materials—line back and worsted face. The face of both is formed by inserting wires between the warp threads in such a manner that on their withdrawal a series of raised loops of the worsted warp is formed, upon which the design appears. In the Wilton the pile is cut automatically in the loom during the process of weav-

ing. The worsted portion of the carpet being exclusively in the warp, the threads of which are of continuous color throughout the piece, each particular color requires a special set of threads, worked in an independent manner by what is technically termed a "frame." This arrangement secures great perfection and clearness of design, for each color is brought to the surface entirely by itself, while the others are carried thru or under the linen back. The work has the appearance of embroidery on canvas. For a more detailed description of carpet weaving, see Encyclopaedia Britannica, 1953, volume 4, pages 917 to 922.

Example

A polypropylene continuous filament yarn is prepared by conventional methods from pelleted polypropylene. The yarn is bulked by the conventional crimping technique, to give a somewhat more resilient and bulky yarn. A skein of continuous yarn is immersed in a bath of allyl methacrylate maintained at 25° C. for about 24 hours to achieve a saturation of about 4 percent by weight. The monomer-saturated yarn is subjected to gamma irradiation provided by a linear accelerator at full power (360 pulses per second), to produce a 3 kilowatt beam of electrons on a tungsten target which converted to electrons to gamma rays. The irradiation is continued for a period of time sufficient to give a total dosage received of approximately 5 megarads. The resulting yarn when incorporated into a conventional carpet structure will give greater resilience under long term load than could be achieved by the prior art methods.

Reasonable variation and modification are possible within the scope of the foregoing disclosure and appended claims.

I claim:

1. A method for providing a carpet of good tuft resilience woven from fiber comprising: homopolymers and copolymers of propylene, which comprises:

- (a) spinning polypropylene continuous filaments from polypropylene resin;
- (b) combining said filaments into continuous filament and staple yarns;
- (c) bulking the resulting polypropylene yarn;
- (d) immersing the bulked yarn in a bath of a polyfunctional monomer at a temperature between 20° and 120° C. selected from the group consisting of divinyl benzene, the mono-, di-, tri- and tetra-ethylene glycol diacrylates, the mono-, di-, tri- and tetra-ethylene glycol dimethacrylates, vinyl acrylate, vinyl methacrylate, allyl acrylate, allyl methacrylate, the divinyl ether of diethylene glycol, diallyl maleate, diallyl itaconate, diallyl malonate, diallyl benzene phosphonate, triallyl phosphate, triallyl cyanurate, glyceryl trimethacrylate;

(e) exposing the monomer-saturated bulked yarn to a field of high energy radiation for a time sufficient to give a total dosage within the range between 0.05 and 50 megarads, thereby cross-linking of the monomer-polymer components thereof; and

(f) conventionally weaving the thus treated polypropylene yarn into a carpet.

2. A method for providing a carpet of good tuft resilience woven from polypropylene fiber comprising:

- (a) conventionally spinning polypropylene continuous filaments from polypropylene resin;
- (b) combining said filaments into continuous filament and staple yarns;
- (c) crimping the resulting polypropylene fiber;
- (d) immersing the crimped fiber in a bath of allyl methacrylate;

(e) exposing the monomer-saturated crimped fiber to a field of high energy radiation for a time sufficient to give a total dosage within the range between 0.05 and 50 megarads and thereby impart resilience to said fiber by cross-linking of the said monomer component; and

(f) conventionally weaving the thus treated polypropylene yarn into a carpet.

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