

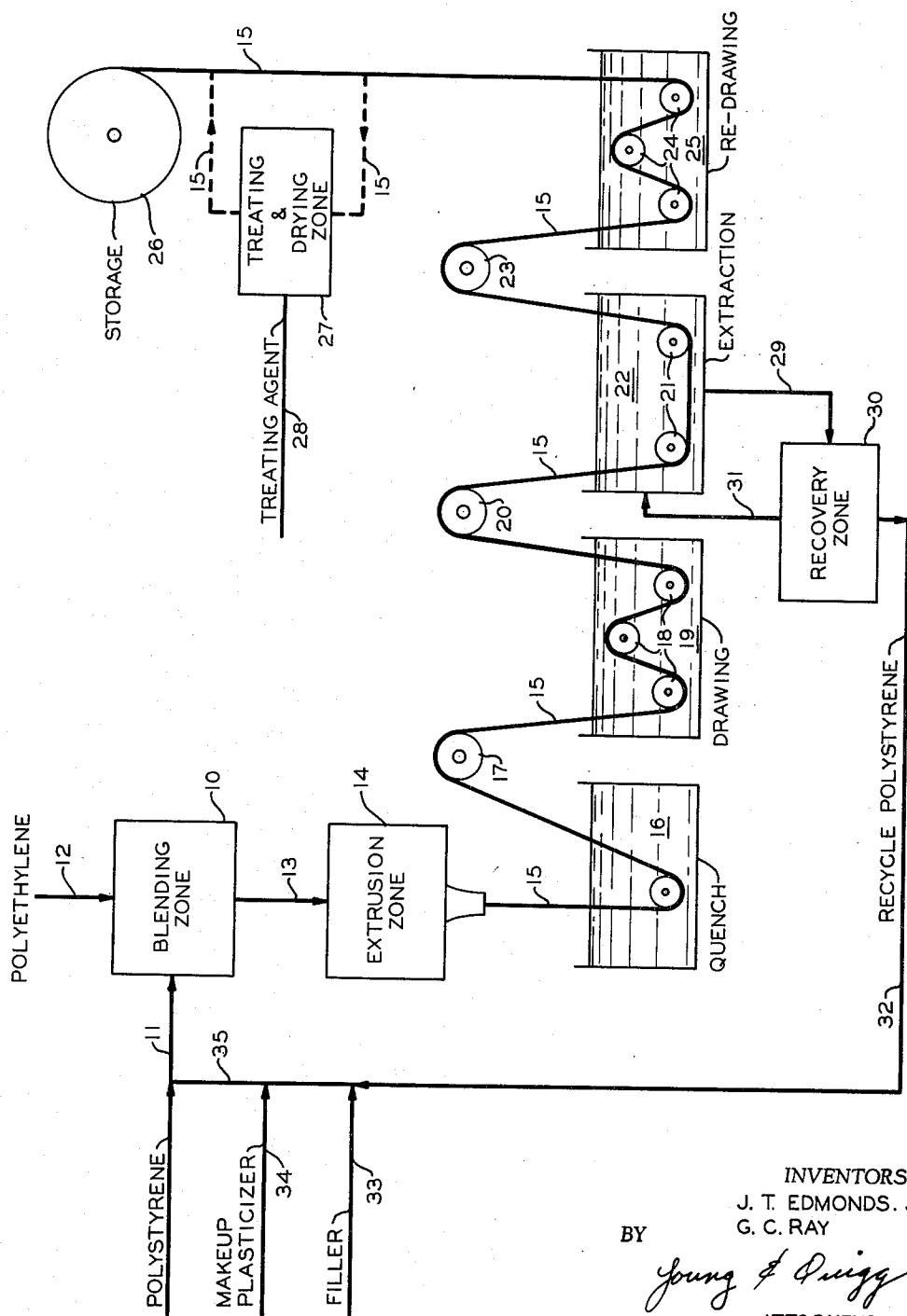
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PREPARATION OF MICROPOROUS FILAMENTOUS ARTICLES

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PREPARATION OF MICROPOROUS
FILAMENTOUS ARTICLES

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This invention relates broadly to microporous filaments or fibers and to filamentous articles formed therefrom. In accordance with one aspect, this invention relates to a method for the preparation of microporous filaments or fibers. In another aspect, this invention relates to novel microporous filaments or fibers and articles formed therefrom.

In recent years, various processes have been developed for the production of polymers of 1-olefins, which polymers have found wide utility in the production of various molded items including filaments or fibers and filamentous articles. Although filaments or fibers formed from 1-olefin polymers have found wide utility, the industry is constantly seeking new uses as well as ways of improving on the properties of these filaments. Filaments formed from high density, solid olefin polymers, especially polyethylene, have excellent tensile properties, chemical resistance and durability. Marine ropes and various wear resistant fabrics have been woven or knitted from these filaments, and many other uses will, no doubt, be found for these filaments in the future because of their inherent resistance to most solvents and other dependable properties of these materials.

The present invention relates to a process for the production of microporous filaments or fibers, which are formed from polymers of 1-olefins, and to the resulting microporous filaments and articles formed therefrom. The porous filaments obtained according to the invention have a softer texture and more crinkle than non-porous filaments, thereby yielding fabrics or other filamentous articles with different qualities and greater insulation properties than filamentous articles made from non-porous filaments.

Accordingly, an object of this invention is to provide novel microporous filaments formed from polymers.

Another object of this invention is to provide a method for preparing microporous filaments or fibers formed from polymers.

A further object of this invention is to provide 1-olefin polymer filaments having improved texture, greater insulating properties, and the like.

Other aspects, objects, as well as the several advantages of this invention will become readily apparent upon a study of this disclosure, the drawing and the appended claims.

According to the invention, novel microporous filaments if improved properties are formed by blending together a 1-olefin polymer and a styrene polymer, extruding the polymer blend into one or more filaments, drawing a filament of said blend to effect orientation of same, extracting the styrene polymer from the drawn filament and thus form a microporous filament, and recovering said porous filament as a product of the process. When desired, the recovered microporous filament can be re-drawn for maximum tensile strength with a corresponding reduction in porosity.

As a further feature of the invention, an additional plasticizer can be employed along with the polystyrene during the formation of the 1-olefin polymer-polystyrene blend. These additional plasticizers can be the high boiling esters including both inorganic and organic acid esters. The added plasticizer is removed from the fila-

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ment along with the polystyrene during the extraction step.

The polyolefins or 1-olefin polymers that can be employed according to the invention include the solid homopolymers of a 1-olefin containing from 2 to 8 carbon atoms, inclusive, as well as copolymers of these 1-olefins with each other. Polyolefins preferred according to the invention include polymers of ethylene and polymers of propylene. Specific representative examples of polyolefins that can be employed include polyethylene, polypropylene, ethylene-propylene copolymers, ethylene-butene-1 copolymers, ethylene-pentene-1 copolymers and the like. Generally, in the case of the copolymers formed by copolymerization of ethylene and a second monomer, the amount of said second monomer present during formation of the copolymers will be less than about ten percent, frequently less than five percent of the total monomers charged to the polymerization zone. Ethylene homopolymers and copolymers of ethylene as described above preferred according to the invention have a density ranging from 0.930 to 0.990, ordinarily 0.940 to 0.970.

The polyolefins or 1-olefin polymers employed according to the invention can be prepared by any of the well known methods which are usually employed in the preparation of these polymers. One well-known method for preparing polymers of 1-olefins is the chromium oxide-catalyzed polymerization described in the Hogan et al. patent, U.S. 2,825,721 (1958). The polyolefins prepared by Hogan et al. are characterized by their high density and high crystallinity.

In addition to the foregoing method for preparing the 1-olefin polymers, especially polymers of ethylene or propylene, the invention also includes within its scope the preparation of microporous filaments prepared from polyolefins formed by polymerization in the presence of an organometal catalyst such as a trialkylaluminum in conjunction with a titanium halide or salt. The polymers resulting from the chromium oxide catalyst polymerization according to Hogan et al., supra, are preferred, and it is further preferred that the very high molecular weight 1-olefin polymers which result from effecting the polymerization at a temperature below that which the polymer dissolves in the reaction diluent (also known as "particle form" polymer) be used.

The polystyrene which is employed in the formation of the blends according to the invention can be a styrene homopolymer or one of the so-called impact styrenes, these being blends of a major amount of polystyrene with a minor amount of rubber, generally of the butadiene-styrene type. It should be understood, however, that any of the high or low molecular weight polystyrenes as well as the impact grades of this polymer can be employed. Any of the several commercially available polystyrenes can be employed in the invention and that polymer which is known in the art as general purpose polystyrene is particularly suitable. The preparation of polystyrene is well known in the art and need not be further discussed herein.

Fibers or monofilaments that can be formed into microporous articles according to the invention ordinarily have a diameter of 0.01 to 100 mils (0.00001 to 0.1 inch), and more generally about 0.1 to 50 mils (0.0001 to 0.05 inch). The tensile strength of these fibers cold drawn is above about 30,000 p.s.i., frequently about 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. Spun threads, cordage rope, woven fabrics and other filamentous articles can be prepared from the microporous filaments of the invention.

As indicated above, plasticizing agents such as the high boiling esters of both inorganic and organic acids can be incorporated into the 1-olefin polymer when blending

same with a polystyrene. Representative specific examples of plasticizers that can be used include the organic acid esters such as diisobutyl phthalate, diamyl phthalate, butyl octyl phthalate, butyl decyl phthalate, di-n-octyl phthalate, diisooctyl phthalate, butylphthalyl butyl glycolate, ethylphthalyl ethyl glycolate, methylphthalyl ethyl glycolate, diethyl phthalate, n-octyl n-decyl phthalate, glycerol phthalate, diphenyl phthalate, (di-tridecyl) phthalate, dibutyl adipate, di-n-hexyl adipate, dicapryl adipate, di(2-ethylhexyl) adipate, diisooctyl adipate, dinonyl adipate, octyl decyl adipate, dibenzyl adipate, diisooctyl azelate, diisobutyl azelate, butyl oleate, tetrahydrofurfuryl oleate, n-butyl palmitate, isooctyl palmitate, dibutyl sebacate, dibenzyl sebacate, octyl stearate, cyclohexyl stearate, glycerol stearate, and the like, inorganic acid esters such as tributyl phosphate, tri-(2-ethylhexyl) phosphate, tri(butoxyethyl) phosphate, triphenyl phosphate, diphenyl octyl phosphate, cresyl diphenyl phosphate, tricresyl phosphate, tri(dimethylphenyl) phosphate, diphenyl o-xenyl phosphate, dibutyl butyl phosphonate, o,o,o-triisooctyl phosphorothioate, and the like.

The amount of plasticizer incorporated into the polymer will generally range from about 5 to about 50 parts per 100 parts of polyolefin/polystyrene blend when no filler is employed. If fillers are employed, the amount of plasticizer can be as high as 100 parts per 100 parts of polymer blend. The preferred range of plasticizer is from 10 to about 40 parts per 100 weight parts of polymer blend. The amount of filler or reinforcing agent employed will ordinarily range from about 1 to about 50 weight parts per 100 weight parts of polyolefin, preferably 2-20 weight parts. Examples of suitable fillers or reinforcing agents that can be compounded into the polymer along with the plasticizer include clay, carbon black, silica, and the like. If pigments are to be used, only very small amounts are necessary to obtain intense coloration of the plasticized polymer.

In forming the blends of 1-olefin polymer with polystyrene, the materials are blended together at a temperature above the softening point of each of these polymers. Generally, this blending step will be carried out at a temperature between about 275 and 500° F., preferably from 300 to 450° F. The specific blending temperature will depend upon the particular polymer being used and the particular type of equipment which is being used to form the blend. Conventional blending equipment such as Banbury mixers, roll mills and the like are quite satisfactory for blending these materials. In the subsequent step wherein a fiber is extruded, a conventional type extruder with a filament die is required. A blending of the polymers in the extruder barrel can be carried out, if desired, although it may be necessary to employ an extended barrel on the extruder to obtain sufficient or adequate blending.

The amount of polystyrene which is blended with 1-olefin polymer can vary over a wide range, but the polyolefin/polystyrene blend will generally contain from 5 to 50 weight percent polystyrene. Higher amounts can be used, but the fibers tend to go to pieces when subsequently extracted. Likewise, lower amounts can be used but one does not obtain any substantial degree of porosity in the fiber.

Following the formation of the blends, the blended material is extruded into filaments by means of conventional fiber or filament forming equipment. This can be carried out with an extruder equipped with a filament die, the die being either monofilament or multifilament. One of the advantages of the present invention is obtained in the extrusion step if one employs one of the very high molecular weight polymers which results from slurry polymerization. These polymers are extremely difficult to extrude or injection mold because of their very low melt index. In general, such polymers have an inherent viscosity in the range between 3.0 and 10.0, and a high load melt index (ASTM D1238-57T—procedure F) in the range between 0.6 and 10.0. The presence of the polystyrene

in the blend serves as a processing aid and sharply increases its extrudability.

A better understanding of the invention will be obtained upon reference to the accompanying drawing which diagrammatically illustrates one preferred embodiment of the invention in the formation of a microporous monofilament.

Referring now to the drawing, polyethylene by way of line 12 and polystyrene by way of line 11 are introduced into blending zone 10. Recycle polystyrene and plasticizer, when used, as well as fillers, dyes, etc., are introduced into blending zone 10 by way of lines 33, 34, 35 and 11. Blending of the polymers and additional added materials is effected in zone 10 in a suitable blending apparatus, such as a Banbury mixer, at a temperature above the softening point of the polymers. However, as indicated above, blending of the polymers can be effected in an extruder when desired. The polyethylene-polystyrene blend obtained in zone 10 is removed by way of line 13 and introduced into extrusion zone 14 wherein the blend is forced through a die forming a strand 15. The extruded strand is passed through water-quench 16, which is usually maintained at a temperature of about 75° F.

Quenched filament 15 is removed from quench 16, passed over roll 17, and then passed through drawing zone 19 having rolls 18. The temperature for cold drawing is ordinarily in the range of about 100 to about 260° F. The temperature is maintained by passing the filament through a bath of polyhydric alcohol such as glycerol while stretching the filament to five to ten times its original length. The filament is drawn in zone 19 to orient the polyolefin molecules and obtain a product of usable properties, e.g., high tensile strength, etc.

The drawn filament is removed from drawing zone 19, passed over roll 20 and then passed through extraction zone 22 having rolls 21. Zone 22 contains a suitable solvent for the polystyrene in the filament as well as the plasticizer, when used. Zone 22 can be any apparatus adapted to provide contact of the filament with the solvent under extraction conditions so as to remove the polystyrene and plasticizer from the filament, thus leaving a porous 1-olefin polymer filament. The preferred extracting agents employed in zone 22 are aromatic hydrocarbons such as benzene, xylenes, and similar aromatic materials. The extraction can be carried out at any temperature from approximately 50 to about 175° F. This upper temperature is the practical upper limit since the cloud point of the polyolefin is approximately 180° F. in most hydrocarbon solvents. Generally, the amount of solvent which is used will be sufficient to completely immerse the filament in the solvent in zone 22. The time required for the extraction will vary over a wide range but will generally range from about 5 minutes to about 100 hours, preferably 0.5 to 5 hours. Extraction zone 22 is sized and so constructed as to provide adequate contacting time of the filament with the polystyrene solvent. As indicated above, the aromatic solvents are preferred since polystyrene is most soluble in these materials. However, any solvent for polystyrene which has little effect on the polyolefin at the above conditions can be used.

Within zone 22, polystyrene and any supplemental plasticizer employed are dissolved out of the filament, thereby leaving a porous filament. The porous filament is then removed from zone 22 and passed over roll 23 and then several alternative procedures can be followed.

As shown in the drawing, the porous monofilament withdrawn from the extraction zone is first passed into a redrawing zone 25 having rolls 24. By redrawing the porous monofilament, a monofilament having a much higher tensile strength is obtained. However, it should be recognized that the redrawing operation reduces the porosity of porous filament 15. Conditions of temperature and draw ratios employed in zone 25 can be substantially the same as employed in zone 19. However,

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ordinary redrawing is carried out at draw ratios ranging from 2 to 1 to 6 to 1, and higher, and temperatures ranging from about 100 to about 250° F. The redrawing operation offers an alternative procedure wherein anti-static agents or other materials can be incorporated into the porous monofilament after extraction and then the filament redrawn to close the pores and trap the additive therein.

Alternatively, redrawn porous monofilament 15 is withdrawn from zone 25 and then either passed directly into storage drum 26 or passed through treating and drying zone 27 and thence to storage roll 26. Treating agents such as antistatic agents, antioxidants, ultraviolet stabilizers and the like can be incorporated into the porous filament in zone 27 by introducing same by means of line 28. As indicated above, treating step 27 can precede the redrawing operation in zone 25. If desired, fillers such as carbon black, graphite, titania, fiberglass and the like, as well as dyes can be introduced into the microporous filament, either before redrawing or after redrawing. However, in most cases, it is more convenient to introduce the fillers and dyes into the polymer before extrusion in blending zone 10. Surprisingly, the porous filaments or fibers obtained according to the invention, after extraction, have more crinkle to them and thus have greater insulating properties when woven into fabrics. The fibers also have a softer texture than unextracted non-porous fibers.

Solvent, extracted polystyrene and supplemental plasticizer, if used, are removed from the base of zone 22 by way of line 29 and passed to recovery zone 30. The aromatic solvent is recovered in zone 30 and returned by way of line 31 to extraction zone 22. Recovery zone 30 can be fractionation, solvent extraction or other suitable separation systems known in the art. Polystyrene and supplemental plasticizer, if used, are removed from zone 30 and returned to blending zone 10 by way of line 32, 35 and 11. Make-up plasticizer, if used, is introduced into recycle line 32 by way of line 34. Filler, for example, carbon black, is introduced into recycle line 32 by way of line 33. Fillers are preferably added to the polymer in the blending zone since they are not removed in the subsequent extraction step.

As indicated above, the porous filaments or fibers of the invention have much lower densities than the same shaped items from untreated polyolefins. By operating in the manner described above, one can obtain the advantages of polystyrene as a processing aid during extrusion, yet by extracting and recycling the polystyrene one can use the polystyrene over and over again with obvious economy. The extracted fibers according to the invention contain practically all of the tensile strength of the unextracted fibers which is particularly surprising in view of the large amount of material removed during the extraction step. Furthermore, the porous filaments are well adapted for treating with agents such as antioxidants, antistatic agents and the like.

The following specific examples are intended to illustrate the advantages of the novel porous fibers or filaments of the invention and the process for their manufacture. However, it is not intended to limit the invention to the embodiments shown in these examples as the examples merely illustrate the various advantages of the invention.

EXAMPLE I

A blend of a high molecular weight polyolefin (ethylene polymer) and polystyrene was formed, spun into a fiber, and extracted.

The high molecular weight polyolefin used in this run was an ethylene/butene copolymer, prepared by polymerization in the presence of chromium oxide-containing catalyst according to the method of Hogan et al., supra, at a temperature such that the polymer, as formed was insoluble ("particle form" polymer) in the reaction diluent. (Polymerization of ethylene-butene-1 is carried

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out in suspension and below temperature at which any substantial portion of the polymer formed is in solution in the suspending medium.) This polymer had the following properties:

Polyolefin Properties

Volatiles	0.11 weight percent.
Ash	0.02 weight percent.
Melt index	1.50.
Density	0.943 g./cc.
Izod impact strength	10.29 ft. lbs./in. notch.
Flexural modulus	150,000 p.s.i.
Tensile yield	3,342 p.s.i.
Elongation	443%.
Environmental stress cracking, time to fail	1,000 hours.
Inherent viscosity	3.71.

The polystyrene used was a commercial polystyrene manufactured by Koppers, sold under the tradename Dylen, having the following properties:

Polystyrene Properties

Density	g./cc.	1.0724
Inherent viscosity		0.84
Hardness, Shore D		84
Tensile strength at yield	p.s.i.	1188

The above described polyolefin (ethylene-butene-1 copolymer) was blended with the above described polystyrene to form a blend containing 75% by weight polyolefin and 25% by weight polystyrene. Blending was carried out in a heated Banbury mixer at 300-370° F. The blend was then spun into a monofilament fiber and drawn at a draw ratio of 6.9 to 1. The initial diameter of the fiber was 0.040 inch, and the drawn fiber diameter was 0.016 inch. The fiber was then soaked overnight in xylene at room temperature, after which the fiber was washed with hot acetone and hot chloroform to remove occluded xylene. After drying under vacuum, the polymer was weighed, and it was found that the extraction had removed 20% of the fiber weight. The extracted fiber was softer to the touch and more pliable than the unextracted fiber. The tensile strength of the fiber, before and after extraction, was as follows:

	Unextracted fiber	Extracted fiber
Tensile at yield, p.s.i.	50,050	49,460
Elongation at break, percent	12	24

In another run, a 90/10 polyolefin (ethylene-butene-1 copolymer)/polystyrene blend was formed from the same polymers as above and extracted by the same method except that the extractions was carried out using benzene at 160° F. instead of xylene at room temperature. The fiber properties (tensile) were as follows:

	Unextracted fiber	Extracted fiber
Tensile at yield, p.s.i.	55,536	49,168
Elongation at break, percent	22	66

The weight decrease of the fiber due to extraction was 9.4%.

EXAMPLE II

In another run, porous fibers were formed by blending high molecular weight polyethylene, polystyrene and di-n-butyl phthalate, spinning the blend into fibers, drawing the fiber and subsequently extracting the thus-formed fibers.

In this run, the polyethylene used was prepared by the polymerization of ethylene in the presence of a chromium

oxide-containing catalyst at a temperature such that the polymer was insoluble in the polymerization medium. This polymer had the following properties:

Polyethylene Properties

Volatiles -----weight percent--	0.0
Ash -----do-----	0.01
Melt index-----	1.87
Density -----g./cc-----	0.955
Izod impact strength-----	No break
Flexural modulus-----p.s.i.-----	209,000
Tensile (yield)-----do-----	3835
Elongation -----percent-----	48
Environmental stress cracking, time to fail-----hours-----	46
Inherent viscosity-----	3.27

The blend was formed by mixing together 80 parts by weight of the above described polyethylene, 10 parts by weight of polystyrene (identical to Example I) and 10 parts by weight di-n-butyl phthalate (DNBP) in a Banbury mixer at 300–370° F. This blend was then spun into fibers and drawn at a draw ratio of 7.0–7.4/1.0. The fiber was then extracted with benzene at 160° F., after which samples of the porous fiber were redrawn by hand at 100° C. The extraction of the original fiber removed 18.1% of the fiber weight. The results of these runs are expressed below as Table I.

TABLE I

Fiber	Diameter, inches	Tensile, p.s.i.	Elonga- tion, percent
Unextracted Fiber-----	0.0160	44,815	90
Extracted Fiber (Porous)-----	0.0160	35,595	115
Redrawn Porous Fiber-----	0.0104	68,240	9
Do-----	0.0080	98,085	10
Do-----	0.0059	113,550	3
Do-----	0.0055	121,850	3
Do-----	0.0050	167,000	4
Do-----	0.0046	210,840	3

EXAMPLE III

Fibers were formed of the blend described in Example II. These fibers were extracted by the method of Example II after which the extracted fiber was redrawn on the fiber machine at a steam box temperature of 210° F. These runs are expressed below as Table II.

TABLE II

Fiber	Diameter, inches	Tensile (yield) p.s.i.	Elonga- tion at Break, percent
Unextracted Fiber-----	0.0128	42,380	103
Extracted Fiber-----	0.0120	49,115	63
Redrawn Extracted Fiber-----	0.0098	95,480	21
Do-----	0.0100	88,740	17

EXAMPLE IV

In another series of runs, a lower molecular weight ethylene/butene-1 copolymer was blended with polystyrene. This blend was extruded into a fiber, after which the fiber was extracted with benzene at 160° F. to remove the polystyrene.

The ethylene/butene-1 copolymer used in this blend was prepared by the solution copolymerization of ethylene and 1-butene in the presence of a chromium oxide-containing catalyst. The copolymer had a melt index (ASTM D1238-57T—Procedure E) of 0.03 and a density of 0.950 g./cc.

This copolymer was blended with the polystyrene described in Example I to form an 85/15 ethylene copolymer/polystyrene blend. The blend was formed by milling the polymer together at 300–370° F. in a Banbury mixer.

The blend was then extruded into fibers, and a portion

of the fiber was extracted with benzene at 160° F. to remove polystyrene. The extraction removed 14% of the polymer weight. The results of the fiber tests are expressed below as Table III. Included in the table are properties of a fiber formed from the ethylene/butene copolymer with no polystyrene present.

TABLE III

Fiber	Fiber Diameter, inches	Tensile at yield, p.s.i.	Elongation at break, percent
Unextracted Fiber containing Polystyrene-----	0.0135	64,800	12
Extracted Fiber-----	0.0138	67,555	35
Fiber from Copolymer Alone-----	0.0130	68,670	12

The extracted fiber from Table III was then redrawn on the fiber machine at a draw ratio of about 2 to 1. The results of these tests are expressed below as Table IV.

TABLE IV

Run	Steam Box Temp. at Draw, ° F.	Fiber Diameter, inches	Tensile at yield, p.s.i.	Elongation at break, percent
1-----	210	0.0130	75,065	27
2-----	230	0.0135	78,820	39
3-----	230	0.0095	90,070	8
4-----	230	0.0105	101,040	11
5-----	230	0.0092	93,730	7

EXAMPLE V

In a further series of testing a blend of polypropylene and polystyrene was formed and extruded into multifilament fiber. The fiber was then extracted with benzene at 160° F. to remove polystyrene.

First, 85 parts by weight polypropylene (Profax made by Hercules—Type 6512-E—commercial extrusion polymer) and 15 parts by weight of the polystyrene described in Example I were blended together at 300–370° F. in a Banbury mixer. The polypropylene polymer employed had the following properties.

Tensile strength-----	4990 p.s.i.
Elongation-----	18%.
Shore D hardness-----	75.
Flexural modulus-----	213,000 p.s.i.
Density-----	0.9066.
Melt index-----	2.8 (measured at 230° C.).

The blend was then extruded into multifilament fiber through a 15 hole die. The fibers were then drawn at 4/1 and 6/1 draw ratios. Portions of these drawn fibers were then extracted for three hours with boiling benzene, after which the fibers were dried overnight. Properties of these fibers are expressed below as Table V.

TABLE V

Sample	Diameter, inches	Tensile, p.s.i.	Elongation, percent
Extrudate (A)-----	0.0140	4,600	510
(A) Drawn at 4/1 ratio-----	0.0065	34,000	22
(A) Drawn at 6/1 ratio-----	0.0050	70,000	16

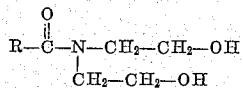
AFTER EXTRACTION

	Wt. Per- cent	Tensile, p.s.i.	Elongation, Percent
Extracted (A)-----	6	49,000	9
(A) Drawn at 4/1 and extracted-----	11.2	33,000	58
(A) Drawn at 6/1 and extracted-----	16.2	48,000	26

EXAMPLE VI

In still another run, 150 grams of 65/35 blend of the ethylene-butene-1 copolymer of Example I/polystyrene was blended with 35 grams of di-n-butyl phthalate and

molded and extracted with benzene at 113° F. for eight hours. The slabs formed were 1/8" x 5 1/8" x 5 1/8". Buffing of the slabs with silk or cotton caused a buildup of static charge of 3.5 to 9.75 kilovolts. One of the slabs was treated with a methanol solution of a commercial antistatic agent sufficient to provide 0.1 weight percent of the agent based on the polymer. The agent used was Alrospers 11-P, an agent sold by Geigy Chemical. Analysis of this agent showed it to be a compound of the formula



wherein R is a 12 to 14 carbon alkyl radical. Testing of the treated slab for static by buffing followed by contact with a voltmeter showed no static buildup. However, in four days, static tests showed a buildup of 7 kilovolts. When the porous polymer was treated with a methanol solution of the agent sufficient to provide 0.6 weight percent of the agent based on the polymer, no static buildup could be effected over a test period of 200 days.

EXAMPLE VII

In still another run, a blend of 50 parts of the ethylene/butene copolymer ("particle form" polymer) of the preceding examples, 50 parts of the polystyrene of the preceding examples and 20 parts of comminuted glass wool was prepared by the procedure of the preceding examples. Molding of a slab at 320° F. and extraction with benzene at 160° F. for four hours produced a porous polymer containing dispersed glass fiber.

Similar runs were carried out using carbon black and TiO₂ as fillers. Porous polymers containing from 5 to 15 percent carbon black were formed, as were porous polymers containing similar levels of titania. Surprisingly, the carbon black did not extract out when the polystyrene was removed by extraction with hot benzene.

The test procedures employed in the preceding examples are set forth below.

Melt index=ASTM D1238-57T—Procedure F unless noted otherwise.

Density=ASTM D1505-60T. Density is run on a compression molded sample, molded at 325 for five minutes and cooled at rate of 27±4° F./minute down to approximately 150° F. (where it can be handled).

Izod impact strength=ASTM D256-56, Method A.

Flexural modulus=ASTM D790-58T.

Tensile and elongation=ASTM D638-58T.

Environmental stress cracking=ASTM D1693-60T.

Inherent viscosity—determined by dissolving an exact amount of polymer (amount will be chosen between 0.02 and 0.1000 gram) in 100 ml. of tetralin at 130° C. The inherent viscosity is calculated as the flow time in seconds in tetralin, both flow times being measured on a size 50 Ostwald-Fenske viscosimeter. From the viscosity, molecular weight can be calculated by

$$\text{Molecular weight} = \frac{K(2.303 \log \eta_r)}{C}$$

where

$K=2.445 \times 10^4$

C =gram of polymer/100 ml. of tetralin @ 130° C.

η_r =inherent viscosity

As will be evident to those skilled in the art, many variations and modifications of this invention can be practiced in view of the foregoing disclosure. Such variations and modifications are clearly believed to come within the spirit and scope of the invention.

We claim:

1. A process for the production of a porous filament which comprises the steps of blending a 1-olefin polymer with from 5 to 50 weight percent of a styrene polymer, shaping said blend into a filament, drawing the filament formed from said blend to orient same, and removing a substantial portion of said styrene polymer from said filament by contacting same with an aromatic hydrocarbon solvent at a temperature ranging from about 50° F. to about the cloud point of said 1-olefin polymer, thus leaving a porous filament of said 1-olefin polymer.

2. A process for the production of a porous filament which comprises the steps of forming a blend of a 1-olefin polymer and polystyrene containing from 5 to 50 weight percent polystyrene, extruding said blend into a filament, drawing said filament formed from said blend within a draw ratio ranging from 2 to 1 to 10 to 1 to effect orientation of same, contacting said drawn filament with an aromatic hydrocarbon solvent under extraction conditions at a temperature ranging from about 50 to about 175° F. and a period of time sufficient to remove a substantial proportion of said polystyrene therefrom, thus forming a porous filament of said 1-olefin polymer, and recovering said porous filament as a product of the process.

3. A process according to claim 2 wherein said porous filament is redrawn within a draw ratio ranging from 2 to 1 to 6 to 1 to form a filament of maximum tensile strength with a corresponding decrease in porosity.

4. The process of claim 2 wherein a high boiling ester is also incorporated into said blend and which is also removed along with said polystyrene during said extraction so as to increase the porosity of said filament.

5. A process for the production of a microporous filament of maximum tensile strength which comprises the steps of blending together a 1-olefin polymer and polystyrene at a temperature above the softening point of the polymers in proportions such that a blend containing from 5 to 50 weight percent of said polystyrene is obtained, extruding the blend thus formed into a monofilament, drawing the filament formed from said blend within a draw ratio ranging from 2 to 1 to about 10 to 1, contacting the drawn monofilament with an aromatic hydrocarbon solvent for said polystyrene at a temperature ranging from about 50 to about 175° F. for a period of time sufficient to extract said polystyrene therefrom and form a porous monofilament, recovering polystyrene extracted during said contacting from said solvent and recycling same to said blending, and recovering said porous filament as a product of the process.

6. A process according to claim 5 wherein said olefin polymer is an ethylene polymer and wherein an organic acid ester is also incorporated into said blend and subsequently extracted along with said polystyrene to increase the porosity of said filament.

7. The process of claim 5 wherein said recovered porous filament is treated with an antistatic agent and then redrawn within a draw ratio of 2:1 to 6:1 to form a filament of maximum tensile strength with a corresponding decrease in porosity, thereby confining a portion of the antistatic agent within the pores of said filament.

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