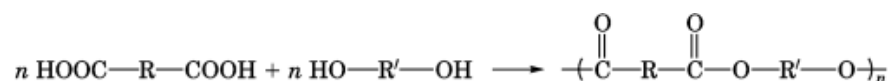
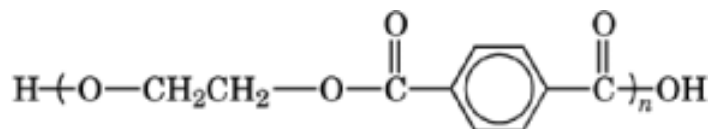


FIBERS, POLYESTER

Polyesters were initially discovered and evaluated in 1929 by W. H. Carothers, who used linear aliphatic polyester materials to develop the fundamental understanding of condensation polymerization, study the reaction kinetics, and demonstrate that high molecular weight materials were obtainable and could be melt-spun into fibers (1–5).



However, because of the low melting points and poor hydrolytic stability of polyesters from available intermediates, Carothers shifted his attention to linear aliphatic polyamides and created nylon as the first commercial synthetic fiber. It was nearly 10 years before J. R. Whinfield and J. T. Dickson were to discover the merits of poly(ethylene terephthalate) [25038-59-9] (PET) made from aromatic terephthalic acid [100-21-0] (TA) and ethylene glycol [107-21-1] (2G).



The Whinfield and Dickson patents (6, 7) dominated the art. The U.S. patent rights were assigned to E. I. du Pont de Nemours & Co., Inc., and Imperial Chemical Industries Ltd. (ICI) obtained the rights for the rest of the world. These patents were quickly followed by patents for improved catalysts for exchange or polymerization reactions (8–12) and for improved fiber properties by drawing (13–15). PET is a fiber of great commercial significance, useful in cordage, apparel fabrics, industrial fabrics, conveyor belts, laminated and coated substrates, and numerous other areas.

1. Properties

The Textile Fiber Product Identification Act (TFPIA) requires that the fiber content of textile articles be labeled (16). The Federal Trade Commission established and periodically refines the generic fiber definitions. The current definition for a polyester fiber is “A manufactured fiber in which the fiber-forming substance is any long-chain synthetic polymer composed of at least 85% by weight of an ester of a substituted aromatic carboxylic acid, including but not restricted to terephthalate units, and para substituted hydroxybenzoate units.”

Poly(ethylene terephthalate), the predominant commercial polyester, has been sold under trademark names including Dacron (Du Pont), Terylene (ICI), Fortrel (Wellman), Trevira (Hoechst-Celanese), and others

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(17). Other commercially produced homopolyester textile fiber compositions include poly(1,4-cyclohexanedimethylene terephthalate) [24936-69-4] (Kodel II, Eastman), poly(butylene terephthalate) [26062-94-2] (PBT) (Trevira, Hoechst-Celanese), and poly(ethylene 4-oxybenzoate) [25248-22-0] (A-Tell, Unitika). Other polyester homopolymer fibers available for specialty uses include polyglycolide [26124-68-5], polypivalolactone [24937-51-7], and polylactide [26100-51-6].

In the late 1980s, new fully aromatic polyester fibers were introduced for use in composites and structural materials (18, 19). In general, these materials are thermotropic liquid crystal polymers that are melt-processible to give fibers with tensile properties and temperature resistance considerably higher than conventional polyester textile fibers. Vectran (Hoechst-Celanese and Kuraray) is a thermotropic liquid crystal aromatic copolyester fiber composed of *p*-hydroxybenzoic acid [99-96-7] and 6-hydroxy-2-naphthoic acid. Other fully aromatic polyester fiber composites have been introduced under various trade names (19).

Most polyester fiber produced is standard molecular weight (ca 0.6 dL/g intrinsic viscosity), round cross-section PET. However, to engineer specific properties for special uses, many product variants have been developed and commercialized. These variants include using alternative cross sections, controlling polymer molecular weight, modifying polymer composition by using comonomers, and using additives including delusterants, pigments, and optical brighteners.

Changing the cross section of standard PET by the use of specially designed spinneret capillaries can change fabric visual and tactile aesthetics. Fabrics with luster and hand ranging from silk or cotton to fur have been made from nonround cross-section fibers (20, 21). Trilobal, pentalobal, octalobal, and scalloped-oval fiber cross sections are currently offered commercially. Fibers containing single or multiple holes are currently used in filling products for improved bulk and thermal management (22–24).

High molecular weight polymer is used for high strength industrial fibers in tires, ropes, and belts. High strength and toughness are achieved by increasing the polymer molecular weight from 20,000 to 30,000 or higher ($DP = 150 - 200$) by extended melt polymerization or solid-phase polymerization. Special spinning processes are required to spin the high viscosity polymer to high strength fiber (25). Low molecular weight fibers are weak but have a low propensity to form and retain pills, ie, fuzz balls, which can be formed by abrasion and wear on a fabric surface (26). Most pill-resistant fibers are made by spinning low molecular weight fibers in combination with a melt viscosity booster (27).

Standard polyester fibers contain no reactive dye sites. PET fibers are typically dyed by diffusing dispersed dyestuffs into the amorphous regions in the fibers. Copolyesters from a variety of copolymerizable glycol or diacid comonomers open the fiber structure to achieve deep dyeability (7, 28–30). This approach is useful when the attendant effects on the copolyester thermal or physical properties are not of concern (31, 32). The addition of anionic sites to polyester using sodium dimethyl 5-sulfoisophthalate [3965-55-7] has been practiced to make fibers receptive to cationic dyes (33). Yarns and fabrics made from mixtures of disperse and cationically dyeable PET show a visual range from subtle heather tones to striking contrasts (see Dyes, application and evaluation).

In addition to dyeability, polyesters with a high percentage of comonomer to reduce the melting point have found use as fusible binder fibers in nonwoven fabrics (32, 34, 35). Specially designed copolymers have also been evaluated for flame-retardant PET fibers (36, 37).

Fibers spun from two different polyesters placed side-by-side or in a sheath–core arrangement have found utility (18, 35, 38, 39). Bicomponent fibers produced from PET and a copolymer can be used as a binder fiber. Bicomponent fibers made from PET and PBT homopolymers are used in apparel applications which take advantage of the dyeability and high recovery of the PBT polyester.

Most textile fibers are delustered with 0.1–3.0 wt % TiO_2 to reduce the glitter and plastic appearance. Many PET fibers also contain optical brighteners (17). Through the use of soluble dyes or pigments, including photochromic pigments (19), a wide variety of producer-colored fibers and effects is available.

Physically or chemically modifying the surface of PET fiber is another route to diversified products. Hydrophilicity, moisture absorption, moisture transport, soil release, color depth, tactile aesthetics, and comfort all can be affected by surface modification. Examples include coating the surface with multiple hydroxyl groups

Table 1. Mechanical Properties of PET Fibers

Property	Staple/tow		Continuous filament		
	Regular tenacity	High tenacity	POY ^a	Regular tenacity	High tenacity
break tenacity, N/tex ^b	0.3–0.5	0.5–0.6	0.2–0.3	0.4–0.5	0.6–0.9
elongation, %	40–60	20–30	110–250	20–40	10–25
elastic recovery, % at 5% elongation	75–80	90		88–93	90
stiffness, N/tex ^b	1–2	5–6	0.2–0.5	1–3	5–7
toughness, N/tex ^b	0.02–0.15	0.02–0.10	0.10–0.20	0.04–0.10	0.04–0.07

^a POY = partially oriented yarn.

^b To convert N/tex to gf/den, multiply by 11.33.

(40), creating surface pores and cavities by adding a gas or gas-forming additive to the polymer melt (41), roughening the surface by plasma treatment of fibers coated with fine particles (42), forming grooves and rough surfaces by combining alkaline treatment with special fiber cross sections (43), and increasing water sorption and improving comfort by alkaline treatment of a freshly extruded fiber surface (44, 45).

1.1. Fine Structural Properties

The performance and properties of PET fibers are significantly impacted by the relative amounts of amorphous and crystalline structures, the orientation of the structures with respect to the fiber axis, and the size distribution of the crystalline regions. By x-ray diffraction, the unit cell of crystalline PET has been determined to be triclinic ($a = 0.456$ nm; $b = 0.594$ nm; $c = 1.075$ nm (46)) with one monomer unit per crystalline unit cell. In the crystalline regions, the molecular chains are almost fully extended (1.075 nm unit cell length vs 1.090 nm theoretical fully extended chain length) with the aliphatic segments in a trans configuration and the aromatic rings in a planar side-by-side register perpendicular to the fiber axis (47).

Density, mechanical, and thermal properties are significantly affected by the degree of crystallinity. These properties can be used to experimentally estimate the percent crystallinity, although no measure is completely adequate (48). The crystalline density of PET can be calculated theoretically from the crystalline structure to be 1.455 g/cm³. The density of amorphous PET is estimated to be 1.33 g/cm³ as determined experimentally using rapidly quenched polymer. Assuming the fiber is composed of only perfect crystals or amorphous material, the percent crystallinity can be estimated and correlated to other properties.

1.2. Mechanical Properties

Polyester fibers are formed by melt spinning generally followed by hot drawing and heat setting to the final fiber form. The molecular orientation and crystalline fine structure developed depend on key process parameters in all fiber formation steps and are critical to the end use application of the fibers.

Molecular orientation and crystallinity generally increase with draw ratio, increasing break tenacity and Young's modulus while decreasing fiber break elongation. Typical properties of continuous filament and staple poly(ethylene terephthalate) fibers are shown in Table 1. Fiber dimensional stability and Young's modulus also are dependent on the heat-setting process. Fibers that are relaxed, or heat-set under no restraint, show a low shrinkage and low initial modulus. Annealed fibers, which are heat-set under tension at constant length, have a low shrinkage and maintain a high initial modulus. Typical stress-strain curves for poly(ethylene terephthalate) fibers are shown in Figure 1. Other factors including polymer molecular weight or the presence of comonomers can significantly affect the fiber mechanical properties.

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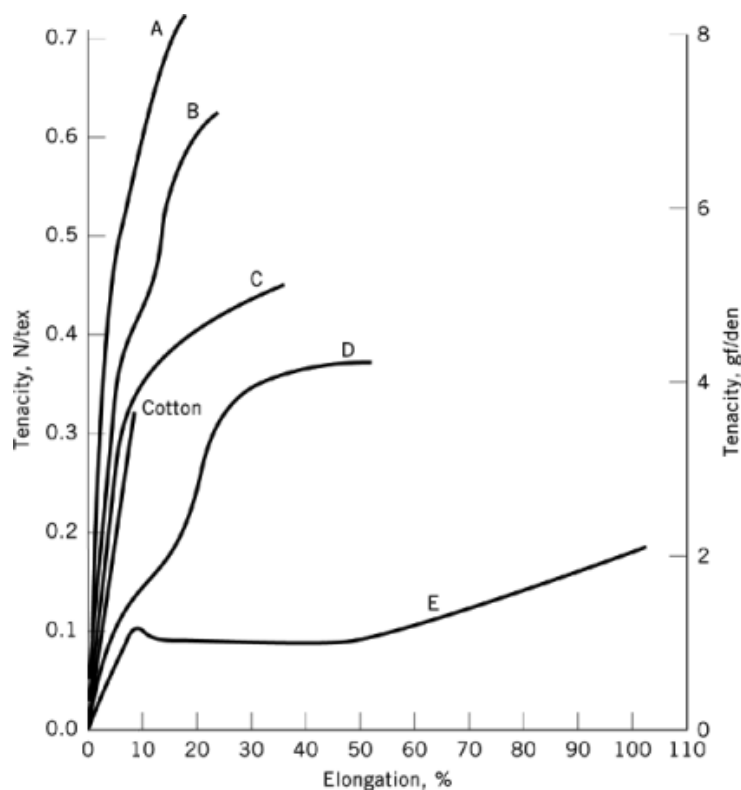


Fig. 1. Typical stress-strain curves for cotton and PET fibers. A, industrial; B, high tenacity, staple; C, regular tenacity, filament; D, regular tenacity, staple; E, partially oriented yarn.

1.3. Chemical Properties

The hydrolysis of PET is acid- or base-catalyzed and is highly temperature dependent and relatively rapid at polymer melt temperatures. Treatment for several weeks in 70°C water results in no significant fiber strength loss. However, at 100°C, approximately 20% of the PET tenacity is lost in one week and about 60% is lost in three weeks (47). In general, the hydrolysis and chemical resistance of copolyester materials is less than that for PET and depends on both the type and amount of comonomer.

At room temperature, PET is resistant to organic and moderate strength mineral acids. At elevated temperatures, PET strength loss in moderate strength acids can be appreciable. Strong acids such as concentrated sulfuric acid dissolve and depolymerize PET.

Polyester fibers have good resistance to weakly alkaline chemicals and moderate resistance to strongly alkaline chemicals at room temperature. PET fibers are attacked by strongly alkaline substances in one of two ways (47). Surface etching is caused by strongly alkaline chemicals including sodium hydroxide (caustic soda) or sodium silicate. Caustic reduction has been used to produce fine diameter fibers from mono- or multicomponent starting materials (17, 47). Other organic bases including ammonia and methylamine penetrate the structure, initiating attack of the polymer molecule in the amorphous regions and resulting in significant loss of strength.

Polyester fibers have excellent resistance to soap, detergent, bleach, and other oxidizing agents. PET fibers are generally insoluble in organic solvents, including cleaning fluids, but are soluble in some phenolic compounds, eg, *o*-chlorophenol.

Table 2. Thermal Properties of PET

Property	Value
melting point, T_m , °C	255–265
glass-transition temperature, T_g , °C	60–77 67 (amorphous) 81 (crystalline)
stick temperature, °C	230–240
heat capacity, C_p , kJ/(kg · K) ^a	
molten polymer (270–290°C)	$1.357 + 2.364 \times 10^{-3} T$
undrawn fiber (–5 to 60°C)	$1.033 + 4.213 \times 10^{-3} T$
thermal conductivity, W/(m · K)	37.5×10^{-3}
thermal diffusivity, cm ² /s	9.29×10^{-4}
heat of fusion, ΔH_f , kJ/mol ^a	24
heat of combustion, ΔH_c , kJ/kg ^a	2.16×10^4

^aTo convert kJ to kcal, divide by 4.184.

1.4. Thermal Properties

The melting point of poly(ethylene terephthalate) is generally reported to be 258–265°C and is generally considered to be independent of molecular weight (17, 47–50). Copolymerization with generated or added comonomers results in a decrease in the melting point and a disruption of the crystalline order (lower crystallinity) dependent on the amount of comonomer present. The melting point of the copolymer can be estimated by:

$$\frac{1}{T_m} - \frac{1}{T_m^0} = \frac{R}{\Delta H_m} \ln n$$

where T_m = melting point of polyester copolymer; T_m^0 = melting point of pure PET polymer; R = ideal gas law constant; ΔH_m = heat of fusion of PET; and n = mole fraction of PET. The heat of fusion of PET is reported (49) to be approximately 24 kJ/mol (30 cal/g) and is dependent on the crystallinity of the polymer. The melting points of most copolymers are 2.5–3.5°C per mol % lower than PET and are independent of the chemical nature of the comonomer.

The glass-transition temperature, T_g , of dry polyester is approximately 70°C and is slightly reduced in water. The glass-transition temperatures of copolyesters are affected by both the amount and chemical nature of the comonomer (32, 47). Other thermal properties, including heat capacity and thermal conductivity, depend on the state of the polymer and are summarized in Table 2.

1.5. Other Properties

Polyester fibers have good resistance to uv radiation although prolonged exposure weakens the fibers (47, 51). PET is not affected by insects or microorganisms and can be designed to kill bacteria by the incorporation of antimicrobial agents (19). The oleophilic surface of PET fibers attracts and holds oils. Other PET fiber properties can be found in the literature (47, 49).

2. Manufacturing and Processing

Terephthalic acid (TA) or dimethyl terephthalate [120-61-6] (DMT) reacts with ethylene glycol (2G) to form bis(2-hydroxyethyl) terephthalate [959-26-2] (BHET) which is condensation polymerized to PET with the elimination of 2G. Molten polymer is extruded through a die (spinneret) forming filaments that are solidified

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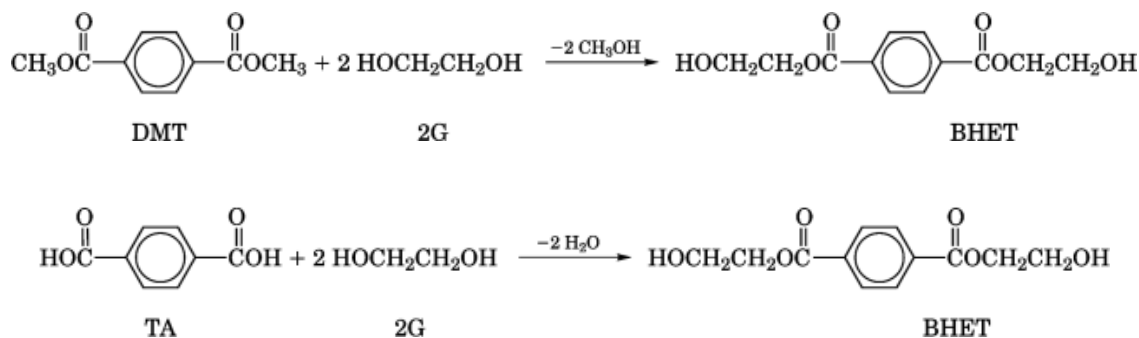
by air cooling. Combinations of stress, strain, and thermal treatments are applied to the filaments to orient and crystallize the molecular chains. These steps develop the fiber properties required for specific uses. The two general physical forms of PET fibers are continuous filament and cut staple.

2.1. Raw Materials

For the first decade of PET manufacture, only DMT could be made sufficiently pure to produce high molecular weight PET. DMT is made by the catalytic air oxidation of *p*-xylene to crude TA, esterification with methanol, and purification by crystallization and distillation. After about 1965, processes to purify crude TA by hydrogenation and crystallization became commercial (52) (see Phthalic acid and other benzenepolycarboxylic acids). In Japan, oxidation conditions are modified to give a medium purity TA suitable to manufacture PET, provided color toners such as bluing agents or optical brighteners are added during polymerization (53). Compared to DMT, advantages of TA as an ingredient are lower cost, no methanol by-product, lower investment and energy costs, higher unit productivity, and more pure polymer because less catalyst is used (54–56). Ethylene glycol is made by oxidizing ethylene to ethylene oxide (qv) followed by hydrolysis (see Glycols, ethylene glycol and derivatives). Catalysts are used in the transesterification reaction of DMT with 2G and in polycondensation. Many compounds have catalytic activity (29). Divalent zinc and manganese are the prevalent transesterification catalysts. Antimony, titanium, and germanium are the predominant polycondensation catalysts. Up to 3% delusterant is added to many PET fiber products to make them more opaque and scatter light; titanium dioxide is the most common delusterant. PET fiber blended with cotton for apparel frequently contains small amounts of fluorescent optical brighteners added during polymerization.

2.2. Polymerization

Commercial production of PET polymer is a two-step process carried out through a series of continuous staged reaction vessels. First, monomer is formed by transesterification of DMT(3) or by direct esterification of TA(4) with 2G:



Starting with DMT, methanol is removed from the reaction; starting with TA, water is removed. Catalysts are used to transesterify DMT but not for direct esterification of TA. The second step is the polycondensation reaction(5) which is driven by removing glycol. A polycondensation catalyst is used.

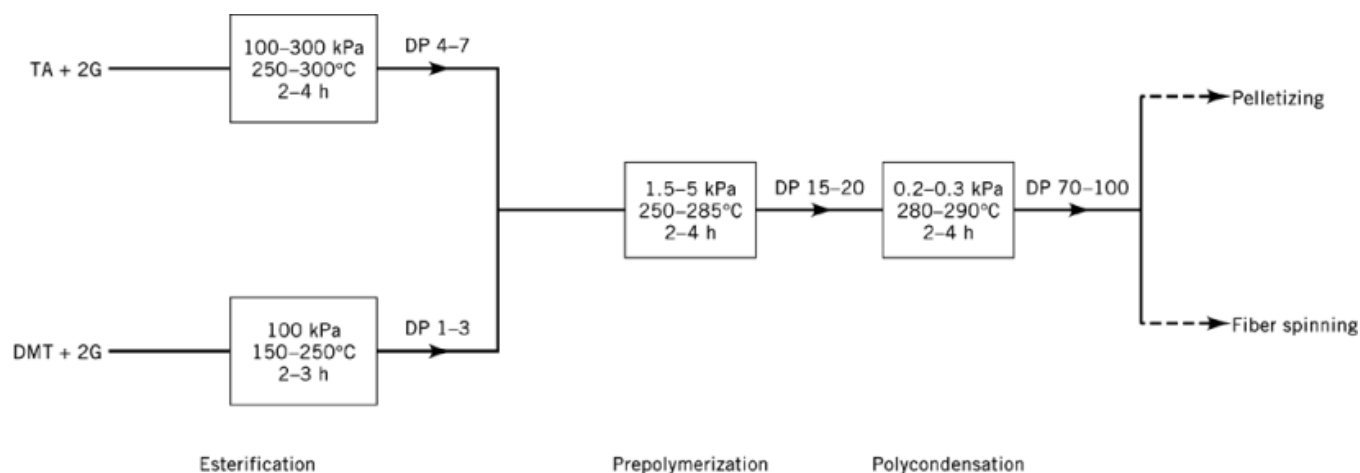
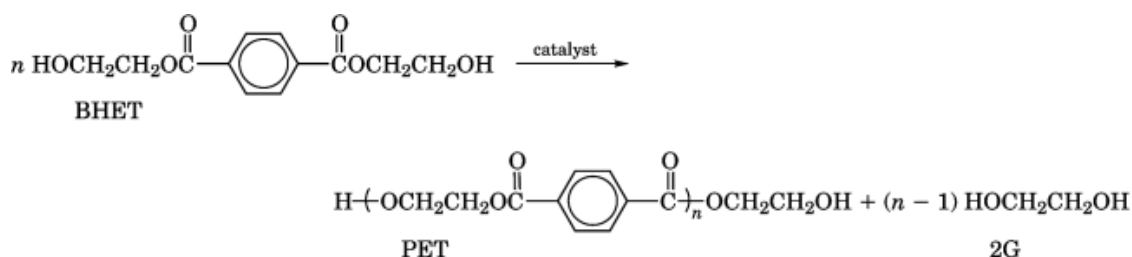


Fig. 2. PET processing conditions. To convert kPa to mm Hg, multiply by 7.5.



In general, esterification is conducted in one or two vessels forming low molecular weight oligomers with a degree of polymerization of about 1 to 7. The oligomer is pumped to one or two prepolymerization vessels where higher temperatures and lower pressures help remove water and 2G; the degree of polymerization increases to 15 to 20 repeat units. The temperatures are further increased and pressures decreased in the final one or two vessels to form polymer ready to spin into fiber. For most products, the final degree of polymerization is about 70 to 100 repeat units. Number average molecular weight is about 22,000; weight average molecular weight is about 44,000. Typical process conditions are shown in Figure 2.

Esterification and prepolymerization vessels may be agitated. Polycondensation vessels have agitators designed to generate very thin films. As PET polymerizes, viscosity increases rapidly to more than 250 Pa·s. Generating surfaces increases the rate of removal of 2G from the melt and, consequently, the rate of polymerization. Agitator designs include screws, ribbons, rotating disks, and wiped films (56, 57). Temperatures and hold-up times are optimized for each set of vessels to minimize the occurrence of side and degradation reactions. By-products in PET manufacture are diethylene glycol, acetaldehyde, water, carboxyl end groups, vinyl end groups, and anhydride end groups (58–60). Oligomers, mostly cyclic trimer, also are present in the range of 1–3% (61). Reaction kinetics are complex; they involve heat and mass transfer and multiple reactions (62–64). Control and removal of the by-products are required to maintain polymer purity and consistency and to protect the environment. Polymer is either pumped directly to fiber spinning units or is solidified and collected as pellets for later remelting and spinning.

For some uses, higher molecular weight polymer consisting of 150–200 repeat units is required. Such polymer usually is prepared by solid-state polymerization in which pellets are heated under an inert atmosphere

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to 200–240°C. The 2G is removed continuously. The rate of polymerization depends on particle size, end group composition, and crystallinity (65).

Older polyester plants generally were based on a batch or semicontinuous polymerization process. Polymer was extruded, solidified in water, and cut to chips or pellets. Capacity was 30 to 60 t/day. In the 1970s and 1980s, polymerization unit capacities increased to 150 to 225 t/day which reduced investment and operating costs per weight of polymer (54, 55). Coupling polymerization and spinning, which eliminated the chipping step, also significantly reduced costs.

2.3. Spinning

PET fibers are made either by directly spinning molten polymer or by melting and spinning polymer chip as shown schematically in Figure 3. A special, precise metering pump forces the molten polymer heated to about 290°C through a spinneret consisting of a number of small capillaries, typically 0.2 to 0.8 mm in diameter and 0.3 to 1.5 mm long, under pressures up to 35 MPa (5000 psi). After exiting the capillary, filaments are uniformly cooled by forced convection heat transfer with laminar-flow air (66). Air flow can be transverse across the bundle, radial from outside-in or from inside-out, or a combination of transverse and radial along the threadline length. Solidification generally occurs from 0.2 to up to several meters from the spinneret (67, 68). Following solidification, the threadline is passed over a finish applicator and collected at speeds of 100 to 7000-plus m/min for subsequent processing. Continuous filament products are small bundles of up to 300 individual filaments. Each bundle is collected on an individually wrapped package for further processing or for direct use. Staple products generally are bundles of 200 to 3000 individual filaments. Each bundle is combined with up to 50 or more others and collected in a large container for further processing. Staple is spun at speeds up to about 2000 m/min. A spin finish is applied to reduce friction and eliminate static.

Flow processes inside the spinneret are governed by shear viscosity and shear rate. PET is a non-Newtonian elastic fluid. Spinning filament tension and molecular orientation depend on polymer temperature and viscosity, spinneret capillary diameter and length, spin speed, rate of filament cooling, inertia, and air drag (69, 70). These variables combine to attenuate the fiber and orient and sometimes crystallize the molecular chains (71).

It is convenient to classify commercial PET spinning processes according to the degree of molecular orientation developed in the spun fiber. Generally, the classification is a function of spinning speed (67, 72): low oriented yarn (LOY) is spun at speeds from 500 to 2500 m/min; partially oriented yarn (POY) is spun at 2500 to 4000 m/min; highly oriented yarn (HOY) is spun at 4000 to 6500 m/min; and fully oriented yarn (FOY) is spun at greater than 6500 m/min. Figures 4 and 5 show some trends in fiber physical properties and fine structure response to spinning speed.

LOY is characterized by low spinning tension, mostly rheological effects, little orientation, amorphous structure, low tensile strength, and high elongation. The spun filament must be drawn, usually three to six times its initial length, and heat-treated before it develops useful properties. Nearly all PET staple is spun this way.

At POY spinning speeds, orientation increases rapidly. Crystallinity begins to increase. The combination gives a fiber of moderate strength and dimensional stability. The discovery of these properties, the development of winders capable of collecting yarn at these speeds, and the discovery of sequential or simultaneous draw texturing led to the explosive growth of textured polyester filament markets in the 1970s and 1980s (73, 74).

At HOY speeds, the rate of increase in orientation levels off but the rate of crystallization increases dramatically. Air drag and inertial contributions to the threadline stress become large. Under these conditions, crystallization occurs very rapidly over a small filament length and a phenomenon called neck-draw occurs (68, 75, 76). The molecular structure is stable, fiber tensile strength is adequate for many uses, thermal shrinkage is low, and dye rates are higher than traditional slow speed spun, drawn, and heat-set products (77).

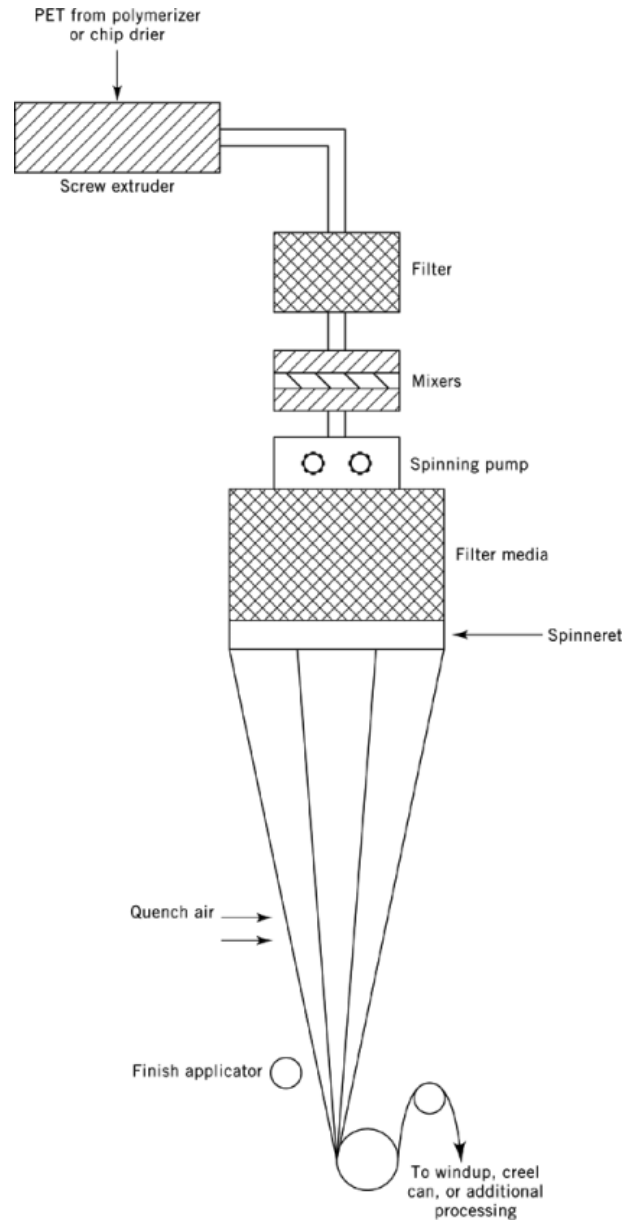


Fig. 3. Schematic diagram of PET spinning.

FOY speeds are the most recent development in PET spinning (78). Properties are similar to HOY and appear to be limited by the differential cooling rate from filament surface to filament core. This leads to radial distribution of viscosity, stress, and, consequently, molecular orientation (75). Fiber tensile strength is limited. Nevertheless, speeds up to 7000 m/min are commercial and forecasts are for speeds up to 9000 m/min by the year 2000 (79). Speeds to 9000 m/min have been studied (68, 80, 81).

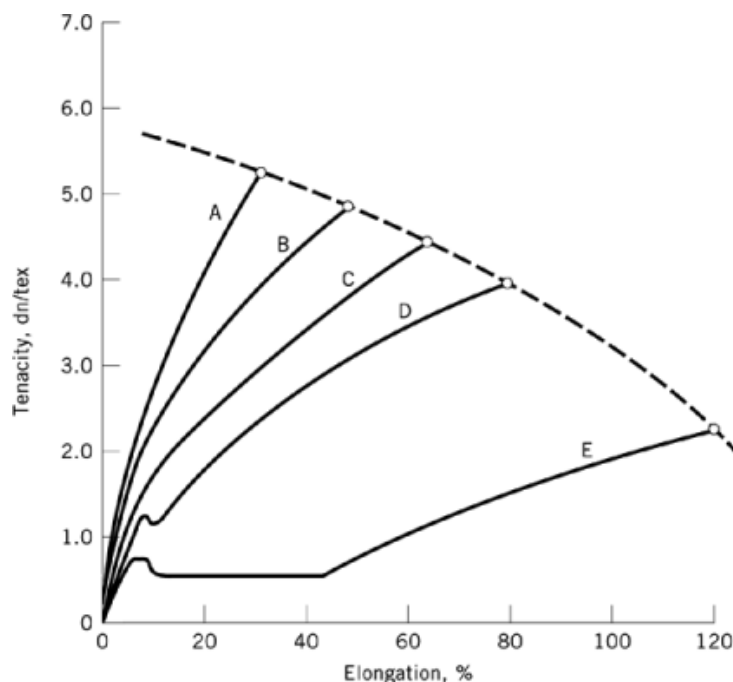


Fig. 4. Representative stress-strain curves of spun and drawn PET: A, low speed spun-mechanically drawn yarn; B, 6405 m/min; C, 5490 m/min; D, 4575 m/min; E, 3202 m/min. To convert dN/tex to gf/den, multiply by 1.13.

2.4. Drawing and Stabilization

Drawing is the stretching of low orientation, amorphous spun yarns (LOY) to several times their initial length. This is done to increase their orientation and tensile strength. The temperature is above T_g , about 80°C, to ensure plastic deformation and maximum elongation after drawing. Molecular chains orient and heat initiates crystallization. Precise control of the temperature profile is necessary to orient the molecular chains before crystallization limits the amount of draw available and the fiber ruptures. Drawing in two or more stages is useful to optimize tensile properties and process continuity (82). Stabilization is heating the fiber to release stress within the molecular chains, melt and reform crystals, and increase the level of crystallinity in order to stabilize the fiber structure. Heating without tension allows the fiber to shrink and reduce orientation. Tensile strength and modulus decrease; thermal dimensional stability and elongation increase. Heating at constant length, with tension, maintains the fiber tensile strength and modulus. Crystallinity increases, but some residual shrinkage remains if the fiber is subsequently heated to above the stabilization temperature. Tensile stress in these structures is borne primarily by the tie molecules bridging the amorphous regions between the crystalline lattices. Consequently, tensile strength is only a small fraction of theoretical bond strengths because tie molecules are a small fraction of the total molecules (69).

2.5. Staple Processes

In staple processing, the containers of combined spun ends are further combined to form a tow band of one to 300,000 tex (2.7×10^6 den) and fed to a large draw line as shown schematically in Figure 6. The tow band is spread out into a flat band tracking over multiple feed and draw rolls. The surface speed of the rolls is from 100 to 400 m/min. Natural staple fibers, such as wool or cotton, have a three-dimensional configuration such

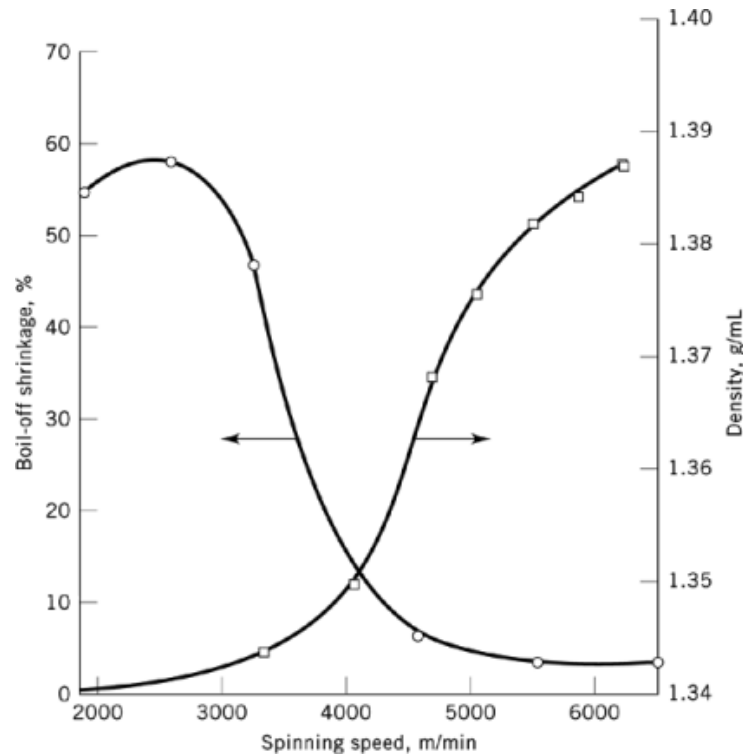


Fig. 5. Density and boil-off shrinkage vs spinning speed.

that groups of them are held together by cohesive forces. Synthetic fibers are not cohesive unless special steps are taken. Crimping is the process by which two-dimensional configuration and cohesive energy is imparted to synthetic fibers so they may be carded and converted to spun yarns. Many proposals have been made to crimp synthetic fibers, but the stuffer box principle dominates commercial operation (69). Two rolls force the tow band into a chamber. Geometrical and frictional restraining forces in the chamber cause the tow band filaments to buckle and form a plug. Crimp frequency, amplitude, and permanence of the bent filament are functions of roll and chamber geometry, frictional restraining forces, and filament temperature. Many staple products are relaxed after crimping under low tension in a continuous-belt convection oven. These products generally are used in applications where high tensile strength and modulus are not critical properties. High strength, high modulus staple fibers are made by passing the flat rope band at high tension over heated multiple rolls (83) or through high pressure saturated steam (84, 85) prior to crimping. Because these fibers have been crystallized in a linear configuration, crimping them is more difficult than crimping products which are later stabilized. The tow band is cut to precise lengths using a radial multiblade cutter, normally 30 to 40 mm for blending with cotton, 50 to 100 mm for blending with wool, and up to 150 mm for making carpets. Cut staple is packaged in up to 500 kg bales at densities greater than 0.5 g/cm^3 and shipped to a mill for further conversion. For some uses, uncut tow is boxed and shipped. Finishes specially designed to facilitate the various conversion processes are applied at one or several stages in the drawing, stabilizing, cutting, and baling processes.

In the last few years, small, 10–15 t/day, compact staple spinning/drawing/cutting/baling units have been offered for plants producing specialty, small-volume products (86, 87). These units offer high flexibility for rapid production changes and simplicity of operation. These units often are used to reprocess polyester polymer recovered from bottles to make carpet fiber or filling products (88).

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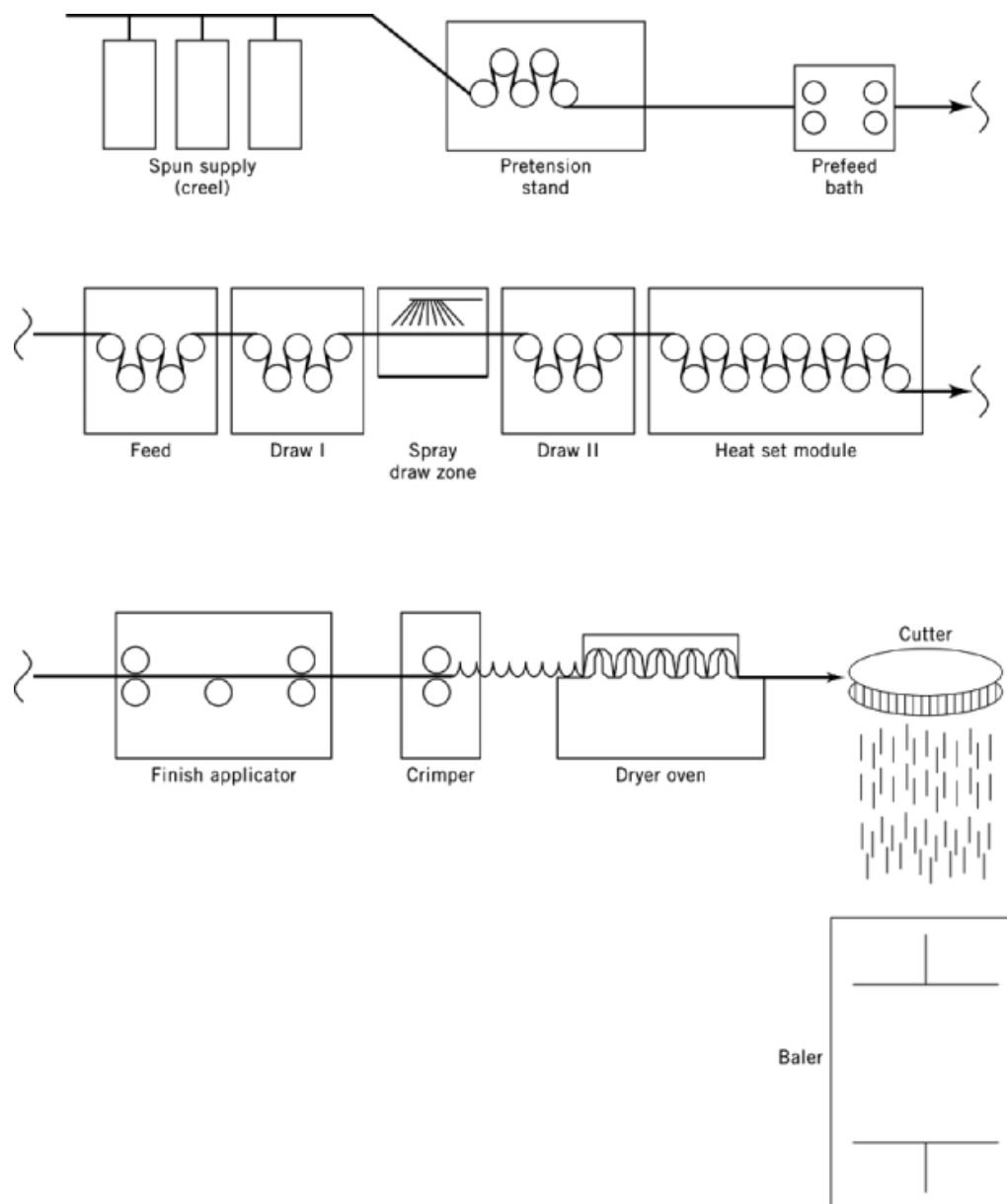


Fig. 6. Schematic diagram of PET drawing.

2.6. Filament Processes

Filament-spun products are generally processed in one of two ways: in the first, spun filaments are drawn and stabilized by passing individual ends over sets of heated rolls to draw, twist or interlace, and stabilize the fiber. Initially, individually packaged ends were unwound, drawn, twisted, and rewound to new individual packages. Later, higher speed processes to draw and stabilize the threadlines during spinning were developed (89). Multiple spun threadlines have been combined into a warp sheet, drawn, and wound up on beams (90).

These products have no crimp. The second process introduces crimp into filament products; the process is called texturing. Its purpose is to increase yarn bulk and elasticity and give the fabric tactile aesthetics more like those from staple. The loops and bends created in the yarn by the texturing process impart fabric tactile aesthetics similar to those created by the free fiber ends in spun staple yarns.

2.7. Texturing

This is a process applied to continuous filament yarns to introduce loops and bends in the individual filaments. The first process to bulk yarn was twisting followed by untwisting and was disclosed in the early 1930s (91). Stuffer box, knit-deknit, false twist devices, and air jets also are used to texture yarns. In the 1970s, with the advent of POY, false twist texturing grew explosively and accounts for most of the textured yarn produced in the 1990s.

In false twist texturing (FTT), shown schematically in Figure 7, a device twists yarn upstream of its location as the threadline passes across a heater, and the yarn untwists downstream from the device and is wound up. If the yarn is not heated downstream from the twist device, it has bulk and high elasticity (stretch). If the yarn is heated downstream from the twist device, it has bulk, but much less stretch. FTT machines initially used two steps to sequentially draw and texture. Later machines combined those steps to simultaneously draw and texture (92). Initial machines used pin spindles as the false twist device. Texturing speeds were about 150 m/min and slowly increased to about 300 m/min. As POY quality and structural stability improved, new friction twist devices have appeared that allow higher speed texturing. Pin twisters control twist directly, but friction twisters control it indirectly by controlling yarn torque. Stacked disks generate rotational friction at the nip points between sets of vertically arranged disks (93). The disks have polyurethane or ceramic surfaces. Crossed belts generate rotational friction between belt surfaces traveling in opposite directions (94). In ring twisters, overlapping disks (rings) rotating in opposite directions generate rotational friction. The yarn path is between the overlapping surfaces of two rings. Normal pressure applied to the ring surface creates frictional twisting forces. The yarn crossing angle, external pressure, and ring-to-yarn speed ratio are key variables (95). Texturing speeds reach up to 1200 m/min. The current barrier to higher speeds is a problem called surging. As speeds and yarn tensions increase, threadline instability caused by intermittent variation in yarn tension develops (96, 97). Texturing speed becomes unstable and yarn quality is unacceptable. Shortening the texturing zone and increasing the rate of heat transfer by using condensing steam are potential routes to increase speeds further (98, 99).

The air jet textured yarn process is based on overfeeding a yarn into a turbulent air jet so that the excess length forms into loops that are trapped in the yarn structure. The air flow is unheated, turbulent, and asymmetrically impinges the yarn. The process includes a heat stabilization zone. Key process variables include texturing speed, air pressure, percentage overfeed, filament linear density, air flow, spin finish, and fiber modulus (100). The loops create visual and tactile aesthetics similar to false twist textured and staple spun yarns.

3. Analytical Test Methods

Physical testing applications and methods for fibrous materials are reviewed in the literature (101–103) and are generally applicable to polyester fibers. Microscopic analyses by optical or scanning electron microscopy are useful for evaluating fiber parameters including size, shape, uniformity, and surface characteristics. Computerized image analysis is often used to quantify and evaluate these parameters for quality control.

Polyester composition can be determined by hydrolytic depolymerization followed by gas chromatography (28) to analyze for monomers, comonomers, oligomers, and other components including side-reaction products (ie, DEG, vinyl groups, aldehydes), plasticizers, and finishes. Mass spectroscopy and infrared spectroscopy can

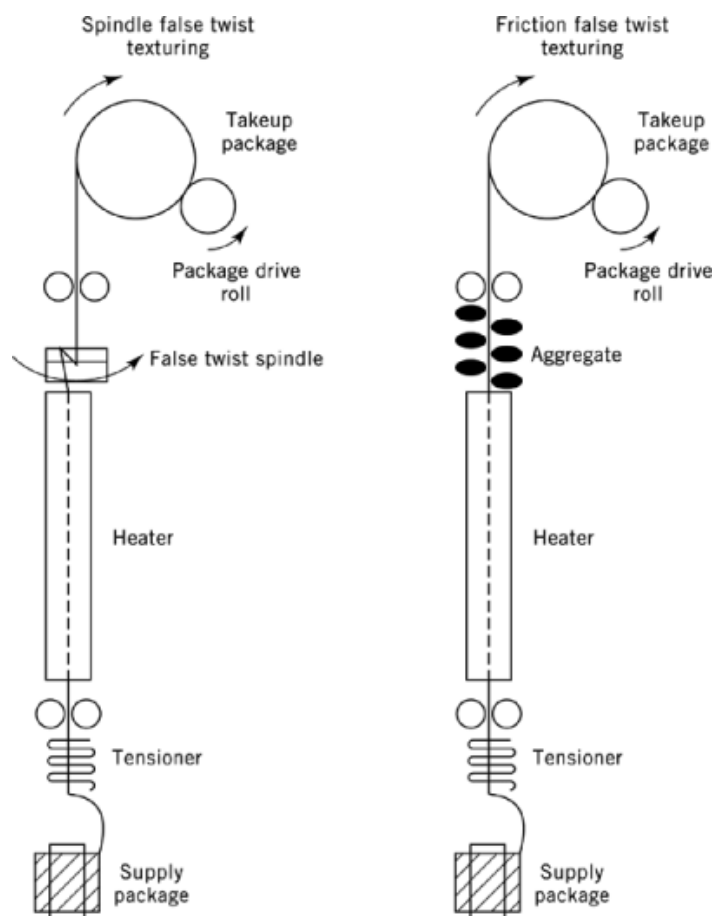


Fig. 7. Schematic diagram of texturing.

provide valuable composition information, including end group analysis (47, 101, 102). X-ray fluorescence is commonly used to determine metals content of polymers, from sources including catalysts, delusterants, or tracer materials added for fiber identification purposes (28, 102, 103).

Gel permeation chromatography can be used to determine the molecular weight and molecular weight distribution of polyester polymers. Polymer molecular weight can also be evaluated using wet chemistry techniques. Polyester polymers are dissolved in strong solvents such as phenol, *o*-chlorophenol, dichloroacetic acid, tetrachloroethane–phenol mixtures, and hexafluoro-2-propanol (17, 47, 49). Relative viscosities, comparing the solution viscosity of polymer solutions at standard concentrations vs the solvent viscosity, are commonly used for quality assurance and control. Intrinsic viscosity, η , is measured from solution and several studies have correlated the number average molecular weight to intrinsic viscosity by a variety of mathematical equations (19, 47, 49, 50).

A variety of analytical techniques have been used to study the structure of polyester fibers. Microscopic refractometry and interferometry can be used to determine the fiber birefringence. Spectroscopic methods including infrared, near infrared, Fourier transform infrared, mass, and Raman spectroscopy have been used to evaluate structural details of polyesters (17, 47, 49, 101–103). Wide angle and small angle x-ray diffraction have been used to obtain information about the crystalline nature of polyesters (17, 47, 49, 102). Thermal

Table 3. 1990 PET Fiber Production Capacity

Manufacturer	Capacity, 10 ³ t	Location
Hoechst	700	United States, Europe
Du Pont	650	United States, Europe
Nan Ya	361	Asia/Pacific
Teijin	352	Asia/Pacific
Wellman	337	United States, Europe
Far Eastern	312	Asia/Pacific
Sam Yang	243	Asia/Pacific

mechanical analysis and nuclear magnetic resonance (nmr) have been used to determine second-order transition temperatures of polyester polymers and fibers. Differential scanning calorimetry (dsc) and differential thermal analysis are useful in detecting thermal properties as well as structural or composition information. Advances in computer control and data acquisition have allowed the on-line monitoring and control of various polymer and fiber parameters such as polymer melt viscosity and fiber spinning tension.

4. Economic Aspects

Since the initial commercial production of polyester fiber at Du Pont's plant in Kinston, North Carolina, in 1953, polyester production expanded to an annual worldwide production of approximately 9.85 million tons in 1992 (104). The growth of worldwide and U.S. polyester fiber production is shown in Figure 8. These data show that although worldwide growth in polyester production has been rapid, polyester production in the United States has been level throughout most of the 1980s. Polyester production has currently leveled in the industrialized countries, with real growth occurring in the developing countries. With several engineering companies designing and installing turnkey operations, polyester production has become a reliable means of creating jobs and supplying feed materials for development of garment-based exports.

Worldwide, the production capacity for polyester fiber is approximately 11 million tons; about 55% of the capacity is staple. Annual production capacity in the United States is approximately 1.2 million tons of staple and 0.4 million tons of filament. Capacity utilization values of about 85% for staple and about 93% for filament show a good balance of domestic production vs capacity (105). However, polyester has become a worldwide market with over half of the production capacity located in the Asia/Pacific region (106). The top ranked PET fiber-producing countries are as follows: Taiwan, 16%; United States, 15%; People's Republic of China, 11%; Korea, 9%; and Japan, 7% (107–109). Worldwide, the top producing companies of PET fibers are shown in Table 3 (107–109).

PET is based on petroleum and the price of polyester fiber fluctuates with the price of *p*-xylene and ethylene raw materials as well as with the energy costs for production. With the ability to interchange with other fibers, especially cotton in cotton blends, the price of polyester is affected by the price and availability of cotton as well as the supply and demand of polyester.

5. Safety and Environmental Factors

5.1. Health Safety

PET fibers pose no health risk to humans or animals. Fibers have been used extensively in textiles with no adverse physiological effects from prolonged skin contact. PET has been approved by the U.S. Food and Drug

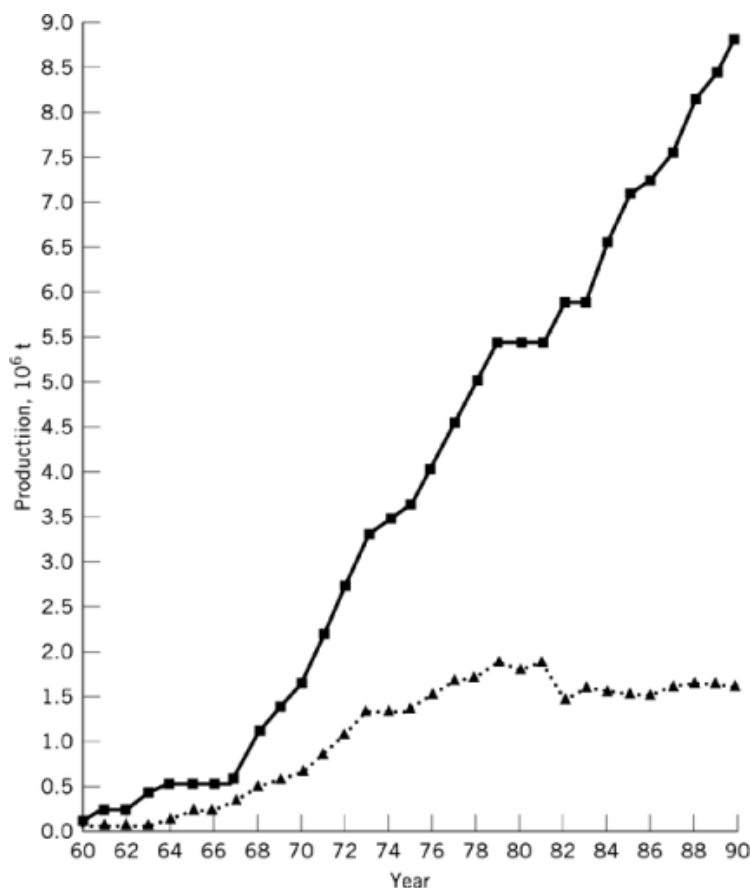


Fig. 8. Worldwide and U.S. PET production, 1960–1990 (—■—), worldwide; (---▲---), U.S.

Administration for food packaging and bottles. PET is considered biologically inert and has been widely used in medical inserts such as vascular implants and artificial blood vessels, artificial bone, and eye sutures (19). Other polyester homopolymers including polylactide and polyglycolide are used in resorbable sutures (19, 47).

When PET is extracted with water no detectable quantities of ethylene glycol or terephthalic acid can be found, even at elevated extraction temperatures (110). Extractable materials are generally short-chained polyesters and aldehydes (110). Aldehydes occur naturally in foods such as fruits and are produced metabolically in the body. Animal feeding studies with extractable materials show no adverse health effects.

5.2. Environmental Factors

PET materials are not dangerous to the environment and cannot contaminate surface or ground water. During polymerization, noncondensable organic by-products are stripped from the process outflow streams and burned. Glycol and water are separated by refining. The water is treated in a standard wastewater facility. The glycol is reused. The methanol from the DMT transesterification is refined and reused. Like all materials, polyesters should be disposed of properly to avoid litter and can be disposed of by landfill or incineration. In sanitary landfills, PET produces no leachate problems and the packing of materials promotes aeration, accelerating

breakdown of biodegradable materials present. In incineration, PET has a relatively high fuel value, ca 23 MJ/kg (9900 BTU/lb), promoting efficient combustion and energy recovery.

A key environmental advantage for PET material is the ability to recycle. Polyester materials, especially bottles, can be separated from contaminated materials such as aluminium caps and paper labels and remanufactured by direct remelt extrusion into fibers for filling products or carpets (111) or into layered constructions for food-grade bottles. In alternative recycling processes, PET can react with methanol (methanolysis) or with ethylene glycol or 1,4-cyclohexane-dimethanol (glycolysis) to produce low molecular weight monomer or oligomers (112, 113) that are recycled back through the polymerization process.

6. Applications

6.1. Staple

PET staple is widely used in 100% polyester or cotton-blend fabrics for apparel. Typical cotton-blend polyester staple fibers have a linear density of about 0.08 to 0.4 tex (0.7 to 3 den) per filament, a tenacity of about 0.4–0.6 N/tex (5–7 gf/den), and a crimp frequency of 3–6 crimps per cm (7–14 crimps per in.). The fibers are coated with about 0.05–0.25 wt % of a finish to reduce friction and control static electricity, cut to about 25–75 mm (1–3 in.), and packaged into 300–500 kg bales. Light, topweight apparel fabrics are commonly 35–65 wt % polyester, and heavier bottomweight fabrics are generally 50/50 blends.

Along with cotton blends, polyester blends with rayon or wool are also important. Wool–polyester blends are widely used in men's suiting materials. For these fabrics, PET staple or tow can be used with a linear density typically about 0.16–0.45 tex per filament (1.5–4 dpf) and a staple length of 50–75 mm (2–3 in.).

In addition to fabrics, PET staple is used in a wide variety of other applications. High tenacity staple fibers are widely used in sewing thread. Staple PET fibers have been engineered for use in rugs, carpets, and filling products including furniture, pillows, mattresses, sleeping bags, and stuffed toys. Polyester staple fibers are commonly used in nonwovens for applications in diaper coverstock, filters, linings and interfaces, and disposable towels and wipes.

6.2. Filament

Fully drawn flat yarns and partially oriented (POY) continuous filament yarns are available in yarn sizes ranging from about 3.3–33.0 tex (30–300 den) with individual filament linear densities of about 0.055 to 0.55 tex per filament (0.5–5 dpf). The fully drawn hard yarns are used directly in fabric manufacturing operations, whereas POY yarns are primarily used as feedstock for draw texturing. In the draw texturing process, fibers are drawn and bulked by heat-setting twisted yarn or by entangling filaments with an air jet. Both textured and hard yarns are used in apparel, sleepwear, outerwear, sportswear, draperies and curtains, and automotive upholstery.

High molecular weight polyester is commonly used to make high strength industrial fibers. Typical yarn bundle sizes of 111–222 tex (1000–2000 den) and single filament sizes of 0.55–1.11 tex per filament (5–10 dpf) are available with tenacities on the order of 0.7–1.0 N/tex (8–10 gf/den). These fibers are commonly used in applications requiring high strength and stability, including tire cord, seat belts, industrial belts and hoses, ropes, cords, and sailcloth.

Polyesters are also used in continuous filament spunbonded nonwovens (see Nonwoven fabrics). Reemay spunbonded fabric is composed of continuous filament PET with a polyester copolymer binder. These spunbonded fabrics are available in a wide range of thicknesses and basis weights and can be used for electrical insulation, coated fabric substrates, disposable apparel for clean rooms, hospitals, and geotextiles (qv).

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