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Title: DuPont Sorona® Polymer: A New Sustainable Platform for Growth

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ABSTRACT

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Demand for Sorona® polymer is growing in fibers, engineering thermoplastics, films, and several other markets. DuPont has licensed Sorona® fiber technology to a number of manufacturers for applications development. This presentation will highlight Sorona® polymer work focused toward engineering thermoplastics.

From corn to polymer to future, Sorona® offers a multitude of advantages. Learn all about the advantages Sorona® offers by visiting <http://www.dupont.com/sorona>

GLOBAL ENVIRONMENTAL CONFERENCE -GPEC 2005
CREATING SUSTAINABILITY FOR THE ENVIRONMENT

Presentation Title

DuPont Sorona® Polymer: A New Sustainable Platform for Growth

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Introduction: Development of sustainable technologies

Several factors arising over the last quarter of the 20th century have led to increased global interest in bio-derived materials and biotechnology.

1. Instability of price and availability of crude oil as a chemical process feedstock
2. Increased attention to recyclability of manufactured products
3. Increased awareness of the relationship between sustainable growth and protecting the environment
4. An added economic stimulus for emission reduction, brought about by the Kyoto protocol.

These factors influence how global manufacturing companies manage themselves for both present and future. An example is E. I. du Pont de Nemours and Company, just starting its third century.

In its latest move towards sustainability, DuPont has set ambitious goals for increased use of renewable resources for both feedstock and energy and reduction of greenhouse gas emissions.

The strategy to meet the first of these targets includes the use of biological processes to make products from renewable raw materials. In support of that strategy, DuPont formed the Bio-Based Materials business (BBM) unit in 2000. BBM's first commercial product is the Sorona[®] polymer, a family of poly(trimethylene terephthalate) (PTT, or "3GT") polymers.

History and Development of Sorona[®] Polymer

Polyester is the most widely used synthetic fiber in the world today in apparel, home furnishings and industrial applications. Since its invention more than 50 years ago, the dominant polyester has been polyethylene terephthalate, also called PET or 2GT. Other varieties of the polymer have been known since its initial development. For example, instead of reacting ethylene glycol with the terephthalic acid ingredient to make 2GT, 1,4-butanediol or 1,3-propanediol can be used to make 4GT or 3GT respectively. The unique properties and attributes of 3GT polymer and fibers have been known for many years. 3GT fibers were reported to have better elastic recovery and lower modulus versus PET and PBT [1]. Until recently, three factors have effectively kept 3GT polymer off the market: the relatively high cost of the 1,3-propanediol; inability to make high quality polymer for downstream end-uses; and more difficult polymer process requirements compared to 2GT.

Since the 1990s, however, significant developments have taken place to improve the business prospects for 3GT. More economical methods have been established for producing 1,3-propanediol ("PDO," also called 3G) in commercial quantities from petrochemical sources. Production of PDO has been demonstrated to be feasible from renewable resources as well, via biological processes [2-5]. Finally, continuous polymerization processes have been refined to enable commercial manufacture of 3GT polymer to "fiber-grade" standards of quality and uniformity. In the last decade, DuPont has generated over 100 patents related to the cost-effective production, processing, and applications of 3GT. A few of those patents are listed at the end of this article.

Unique Properties of Sorona[®] Polymer

Molecular structure and effects on mechanical properties [1,6,7]

Sorona[®] polymer is one member of a family of polymers based on fiber-grade 1,3-propanediol. It is a linear semi-crystalline polymer with a melting temperature of ~228°C and a glass transition temperature of about 50°C. The structure of 3GT polymer is shown in Figure 1.

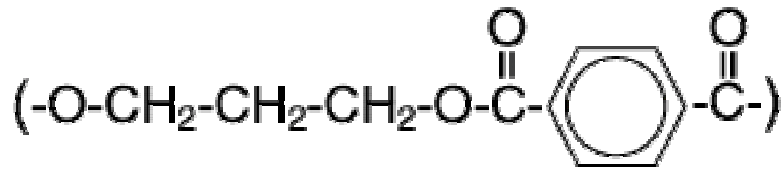


Figure 1. Molecular structure of 3GT

The structure of PTT has been studied extensively [3-5]. The beneficial properties of Sorona[®] polymer are derived from a unique, semi-crystalline molecular structure featuring a pronounced “kink,” as shown in Figure 2.

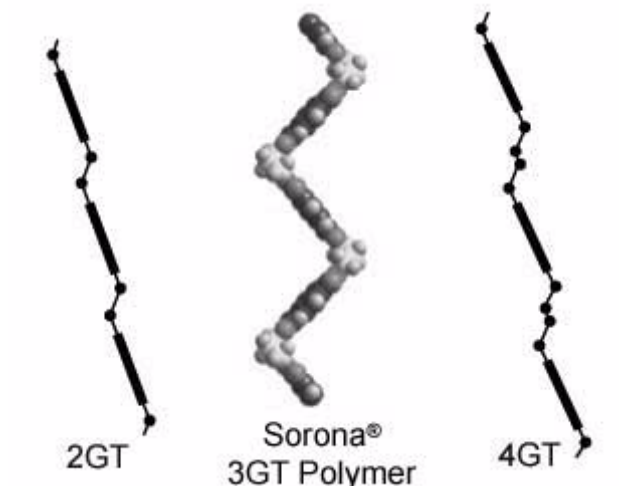


Figure 2. Molecular shape and crystal structure of 3GT

The 3GT polymer shape is a consequence of the convolutions of the bonds in the trimethylene constituent. This zigzag shape means that tensile or compressive forces translate at the molecular level to bending and twisting of bonds, rather than simply stretching. This is analogous to the tensile behavior of a coil spring compared to a straight wire.

When the polymer is cooled from a liquid state, it forms crystalline and amorphous regions. The crystalline regions contribute most significantly to the elastic properties of the solidified polymer. With 3GT, the modulus of the crystalline regions is significantly lower than that of other commercial polymers, e.g., PET (see Figure 3). The fiber can take an appreciable level of applied strain (up to 15 - 20%), and recover completely, e.g., no permanent set, when forces are removed (see Figure 4).

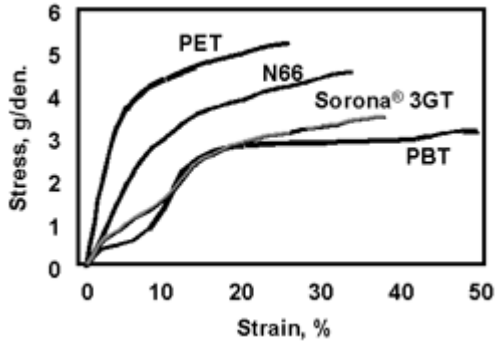


Figure 3. Comparison of tensile characteristics

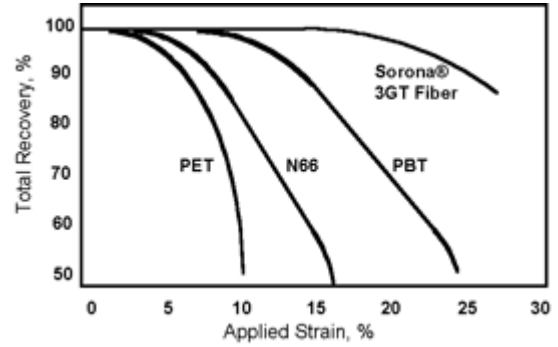


Figure 4. Comparison of stretch recovery characteristics

Table 1 compares properties of Sorona with other synthetic and natural fibers and with polylactic acid (PLA), another bio-derived polymer.

Table 1. Polymer and Fiber Properties

Fiber Property	Synthetic Fibers				Melt spinnable Natural Based		Natural Fibers			
	Nylon 6,6	Nylon 6	PET	Acrylics	Sorona	PLA	Rayon	Cotton	Silk	Wool
Specific Gravity	1.14	1.14	1.39	1.18	1.33	1.25	1.52	1.52	1.34	1.31
Tg (°C)	40-55	40-60	70-80		45-55	55-60	-	-	-	-
Tm (°C)	265	220	260	320 (degrades)	228	130-175	None	None	None	None
Tenacity (g/d)	6 - 10	5.5	6.0	4.0	4-5	6.0	2.5	4.0	4.0	1.6
Moisture regain (%)	4.0	4.1	0.2-0.4	1.0-2.0	0.2-0.3	0.4-0.6	11	7.5	10	14-18
Elastic Recovery (5% strain)	89	89	65	50	100	93	32	52	52	69
Refractive Index	1.54	1.52	1.54	1.50	1.57	1.35-1.45	1.52	1.53	1.54	1.54

- Sorona polymer is currently produced using a chemical process. Full scale commercialization of bio-derived polymer is expected by middle of 2006.

Data for PLA is taken from "Fibers and Fabrics Properties Comparison," downloaded 8/15/2003 from Dow/Cargill website, http://www.cargilldow.com/ingeo/applications_apparel.asp. Data for fibers other than 3GT and PLA are widely published, e.g., *Handbook of Fiber Chemistry*, Lewin, M., and Pearce, E.M., Eds., 2nd Edition, Marcel Dekker Inc., New York, 1998

In addition to its unique stretch-recovery characteristics, Sorona provides all the advantages generally associated with polyesters, i.e., excellent physical and chemical properties, dimensional stability, low moisture absorption, easy care, good weather resistance, easy processability and recyclability. Sorona® polymer can be easily modified to achieve desirable functional properties as well.

Other properties

Sorona® polymer offers several advantages over both conventional polyester (PET) and nylon. It can be effectively disperse-dyed at 100°C eliminating the need for carriers or pressurization in the dyeing process. Once dyed, the fabric exhibits deeper shades and superior washfastness over other products.

Other advantages of Sorona become more evident in actual use conditions. The fiber is highly resistant to most stains without the need for surface treatment with additives or coatings. It resists UV degradation better than other fibers, and exhibits both low water absorption and low electrostatic charging.

Environmental Footprint

The world is increasingly looking at the environmental consequences of a product throughout its life, from raw material through ultimate disposal (see Figure 5). In this area, Sorona® polymer has a number of advantages over other polymers.

- Total LCA energy used in the manufacture of bio-PDO (cradle to grave) is about 40% less than for petrochemically derived PDO.
- The overall 3GT polymerization process is more energy efficient than PET.
- Polymerization and downstream processing of Sorona® polymer saves energy compared to PET due to lower temperatures required, both for processes involving remelt and for dyeing.
- Recycling of Sorona is made much easier by the absence of heavy metals in the product, compared to PET and Nylon.

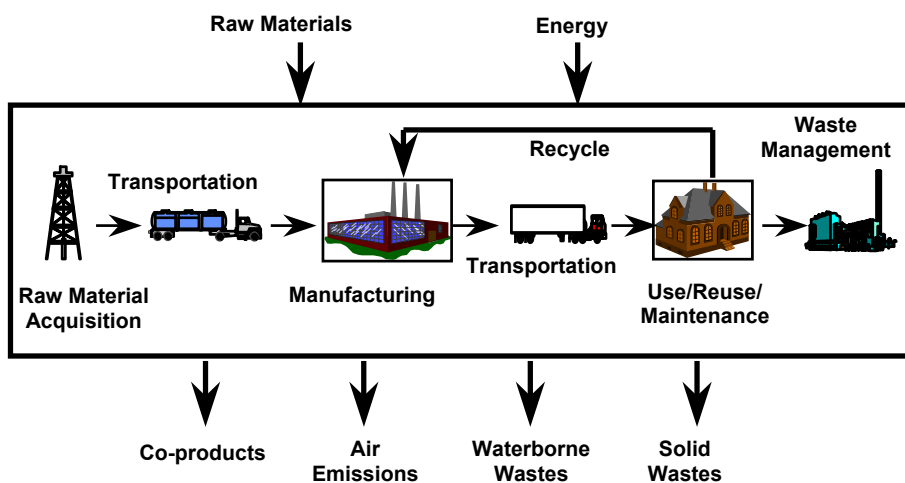


Figure 5. Product Life Cycle: Environmental Effects

PDO and Bio-PDO

Sorona® polymer is made from 1,3-propanediol (PDO) and either dimethyl terephthalate (DMT) or terephthalic acid (TPA). Other materials used in the manufacture of 3GT include an organo titanium catalyst [10-12, 16], and a delustrant (TiO₂).

PDO is a colorless, odorless liquid. The basic properties of PDO are summarized in Table 2, and a typical product specification for polymer manufacture is shown in Table 3.

Table 2. Basic Properties of 1,3-Propanediol (PDO)

CAS number 504-63-2
Chemical structure, HOCH ₂ CH ₂ CH ₂ OH
Chemical formula C ₃ H ₈ O ₂
Molecular weight 76.1
Viscosity at room temperature 52 cp
Specific gravity 1.05
Boiling point 214°C
Freezing point -32°C
Refractive index 1.4386

Table 3. Specific Analytical Properties of 1,3-Propanediol (PDO)
Used for Polymer Manufacture

Purity (GC) 99.9%
 Color (APHA, ASTM D1209) <5
 Water content 0.05% max.
 Ash content <0.001% by weight.

PDO from traditional (chemical) sources [8, 9]

Development of Sorona[®] polymer took place within a business model that requires the commercial success of a product to be established independently of environmental or sustainability considerations. Consequently, early product development and commercialization were undertaken with PDO made from conventional chemical processes, two examples of which are listed below.

A. The Degussa process consists of the following three steps:

1. Oxidation of propylene to acrolein

$$[\text{CH}_2=\text{CHCH}_3 + \text{O}_2 \rightarrow \text{CH}_2=\text{CHCHO}]$$
2. Selective hydration to 3-hydroxypropionaldehyde (3-HPA)

$$[\text{CH}_2=\text{CHCHO} + \text{H}_2\text{O} \rightarrow \text{OCHCH}_2\text{CH}_2\text{OH}]$$
3. Catalytic hydrogenation to 1,3-propanediol (PDO)

$$[\text{OCHCH}_2\text{CH}_2\text{OH} + \text{H}_2 \rightarrow \text{HO}(\text{CH}_2)_3\text{OH}]$$

B. Shell Chemical uses a two-step process:

1. Hydroformylation of ethylene oxide to 3-HPA.

$$[\text{H}_2\text{C} \begin{array}{c} \diagup \text{O} \diagdown \\ \text{---} \end{array} \text{CH}_2 + \text{CO} + \text{H}_2 \rightarrow \text{OCHCH}_2\text{CH}_2\text{OH}]$$
2. Catalytic hydrogenation to 1,3-propanediol (PDO)

$$[\text{OCHCH}_2\text{CH}_2\text{OH} + \text{H}_2 \rightarrow \text{HO}(\text{CH}_2)_3\text{OH}]$$

Bio PDO [2-5]

The need for development of a biological source/process for PDO grew out of several factors:

- Difficulty and cost of producing “polymer and fiber-grade” PDO
- Sustainability of a renewable feedstock vs. a non-renewable feedstock
- The demonstrated efficiency of early trials of bio PDO processes, which showed the potential to be economically competitive with established processes.
- The commitment of DuPont to integrated science, which includes biological science and biotechnology
- The company’s unique position to integrate biotechnology with its longstanding competency in polymer and fiber technology

Initial development of biologically manufactured PDO has been with corn sugar, well known as a plentiful and inexpensive raw material. Conversion of glucose to PDO has been known to occur in nature in two stages: first by yeast to an intermediate product, glycerol, then by bacteria to PDO or 3G (Figure 6).

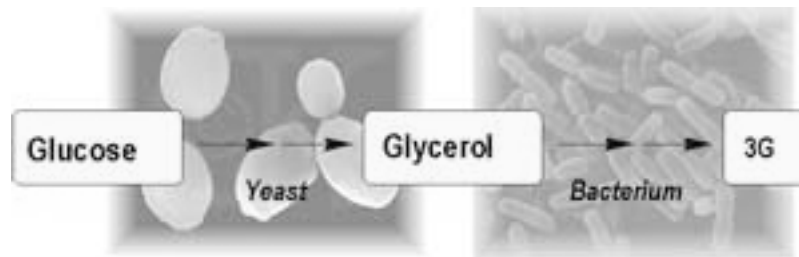


Figure 6. Conversion of glucose to PDO occurs in two steps in nature

DuPont and Genencor International have jointly developed a bacterium (“biocatalyst”) to do both steps in a single fermentation stage. DuPont and Tate & Lyle, a major corn based products company with expertise in fermentation processes, partnered to develop the manufacturing process based on this biocatalyst. The process is represented in Figure 7.

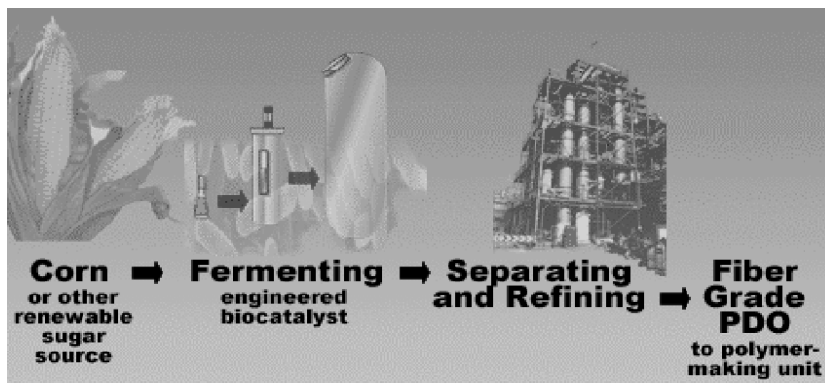


Figure 7. Pictorial representation of process: Corn to PDO

The trial product was confirmed to have attributes and quality equivalent or superior to chemically produced PDO. For example, the results of purity tests of bio based PDO and conventional chemical (Wesseling) PDO are shown in Figure 8. In summary, the percentage of impurities in Bio PDO is around one-tenth the amount of impurities in chemical PDO (0.003% vs. 0.032%).

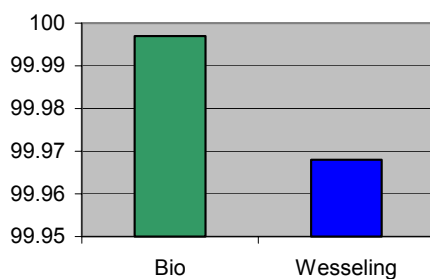


Figure 8. Purity comparison of Bio and Chemical (Wesseling) PDO.

Polymer Manufacture [10-14]

The DuPont 3GT polymerization process technology was developed as a retrofit for existing 2GT facilities. One such converted facility is located at the DuPont site at Kinston, NC, USA, which has been making commercial quantities of 3GT polymer for several years. A schematic diagram of a typical Continuous Polymerization process is shown in Figure 9.

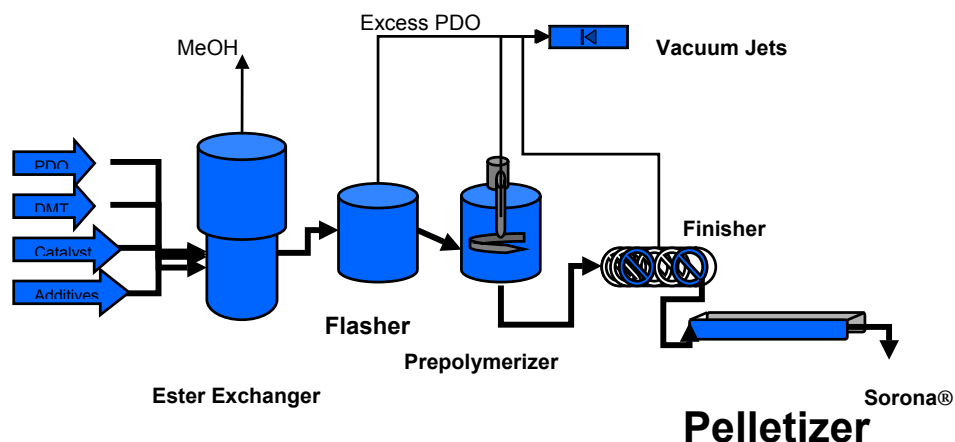


Figure 9. Continuous Polymerization Process

Typical properties of CP polymer, as reflected in the current DuPont specification, are shown in Table 4.

Table 4. Properties of continuously polymerized 3GT
 Product: Sorona[®] polymer (a trademark of E. I. du Pont de Nemours and Company)
 Type: Semi-dull Homopolymer:
 Process: Continuous Polymerization (CP)
 Specification date: March 30, 2001

Property	Aim	Limits		Test Method
		Min	Max	
Intrinsic Viscosity	1.02	1.01	1.03	TM-0590-91
Color, Hunter b	-	5	8	GEN-07
Color, Hunter L	-	80	-	GEN-07
COOH, $\mu\text{eq/g}$	-	-	15	TM-1145-91
Melting Point, deg C	228	224	232	SP-509
TiO ₂ , wt%	0.30	0.27	0.33	TM-1090-90
Melt Flow Index				
Moisture, ppm (as packaged)	-	-	500	SP 525
Pellet Size, wt. of 50 pellets, g	1.6	1.4	1.8	GEN-08
Appearance	No foreign substances, dust, or gross particles			Visual
Package Description	Lined Gaylord boxes, 1475 lbs (670 kg) nominal per box			

Molecular weight analysis of a representative sample from commercial production is given below. Analysis is by SEC triple detection, using HFIP solvent. The refractive-index detector chromatogram is shown in Figure 10 below.

Number average molecular weight $M_n = 30,300$

Weight average molecular weight $M_w = 56,300$

Z average molecular weight $M_z = 85,900$

$M_w/M_n = 1.86$

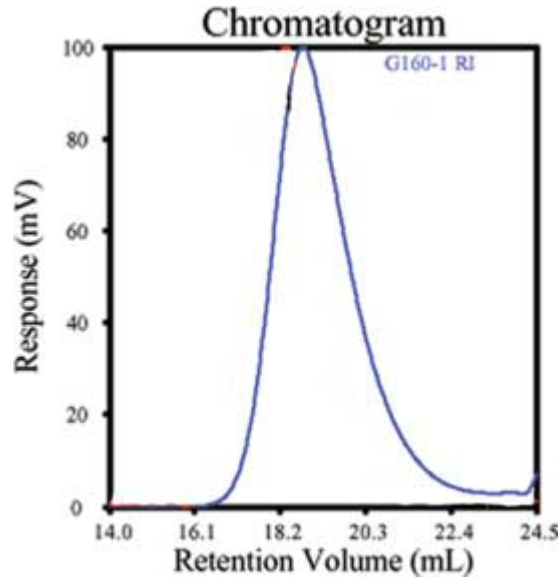


Fig. 10. Size Exclusion Chromatogram (SEC) of production sample of Sorona® polymer (*RI detector*)

Melt Viscosity Characteristics

The behavior of Sorona® polymer in the melt state is similar to other condensation polymers. That is, at low shear rates the viscosity is nearly constant (Newtonian) and at higher shear rates viscosity decreases with increasing shear rate approaching a power-law relationship. Viscous behavior measurements for a melted production sample of Sorona® polymer are shown for several melt temperatures in Figure 11.

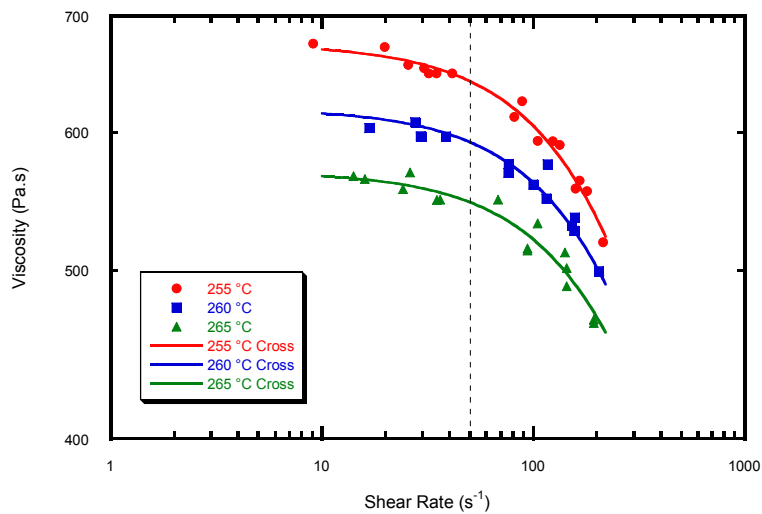


Figure 11. Melt viscosity traces of production sample of Sorona® polymer

Polymer Processing

Remelt

As with most polymers, 3GT is shipped from production facilities in the form of pellets, and delivered to customers where they are remelted to molten polymer and cast, spun, or otherwise processed into intermediate and end-use products.

Because remelt and processing performance of 3GT polymer is more sensitive than 2GT to moisture and impurities, special effort must be made to ensure 3GT is clean, dry, and free of other polymers. Pellets must be dried to <40 ppm moisture before remelting. A nitrogen environment is recommended, although not essential, for drying.

Polymer shaping

Sorona[®] polymer can be converted into a variety of products by conventional methods on existing equipment. The significant differences of Sorona in processing are lower melt temperature (comparable to nylon 6 or polypropylene), lower modulus, higher stretch, and better stretch recovery.

Fibers – apparel/carpets

Lower melt temperature compared to 2GT and nylon 6,6 allows spun-dyed yarn with a greater variety of pigments/colors. 3GT fiber can be spun on “short stack” spinning machines originally built for nylon 6 and polypropylene bulk continuous filament (BCF) yarns. [17-19] Draw [20], windup, texturing [21], weaving, knitting, and tufting performance are comparable to PET once the equipment is properly adjusted for the mechanical properties of 3GT.

Films

3GT can be cast into films at settings comparable to polypropylene or Nylon 6. Process optimization is required to eliminate film brittleness of cast films. Biaxially oriented films can also be prepared using 3GT. Moreover, the 3GT polymer can be modified (e.g., copolymerized with isophthalic acid) or blended with other commercially available polymers (2GT, 4GT, etc.) to make films with a variety of properties.

Nonwovens

The suitability of Sorona® polymer has been demonstrated for various nonwoven processes including flash spinning [23].

Engineering components

All conventional thermoplastic formation techniques can be used with 3GT: injection molding, pressure molding, blow molding, casting, rotary molding, etc. with attention paid to minimize melt time. Otherwise, molding processes are comparable to Nylon 6, PBT, PET, or polypropylene [24]. 3GT molding conditions are similar to that of PBT.

Product Stewardship

Solids Handling

Potential hazards associated with Sorona® polymer handling include inhalation of dusts and thermal burns from contact with hot polymer. Sorona® polymer will typically contain <200 ppm dust by weight. This dust is non-hazardous based on animal exposure tests. Thermal burns from hot polymer should be prevented similar to other polymers (such as PET and nylon).

Polymer Processing

During drying and processing of Sorona® polymer, vapors are emitted which may contain traces of acrolein and allyl alcohol. With proper processing and engineering controls, acrolein and allyl alcohol emissions can be managed to ensure that concentrations do not exceed safe limits.

Holding processing temperatures below 290°C, and preferably below 260 - 265°C, minimizes thermal and thermo-oxidative degradation of Sorona® polymer and consequent formation of acrolein and allyl alcohol. Inert gas blanketing of critical process locations (e.g., extruder throat) will reduce thermo-oxidative degradation.

Additional information on monitoring, control, and disposal of all byproducts; including acrolein and allyl alcohol, is available from DuPont, OSHA, and other sources.

DuPont has patented several technologies to control byproducts during the manufacture and processing of Sorona® polymer.

Applications and End-uses

In end-use performance, Sorona[®] polymer exhibits mechanical properties comparable to or in some cases better than nylons, (e.g., nylon 6) in combination with chemical properties equal to or better than PET.

Fibers – apparel/carpets

The most significant advantages of Sorona fiber for apparel are softness and natural hand, printability and easy dyeability. Additionally, resistance to chlorine and UV add value in outdoor/sport markets. 3GT fibers can be blended with other natural (e.g., cotton, wool, etc.) or synthetic (e.g., PET, acrylics, etc.) fibers for enhanced softness, stretch recovery and other functional attributes.

For floor coverings, Sorona can be made in a variety of colors and styles with good dye uniformity. It offers superior bulk, resilience, texture retention, stain resistance, easy dry, and a softer feel. They're resistant to fading in the presence of UV or chlorine, and they have low electrostatic properties. 3GT is as easy to recycle as PET polyester.

Films

Sorona[®] polymer, either as-is, modified (e.g., copolymers with isophthalic acid, etc.) or blended with other polymers (2GT, 4GT, etc.) can be successfully used in films to obtain desirable property advantages and increased value. For example, a combination of properties of 3GT (oxygen and water vapor barrier, printability and heat sealability) provides desirable attributes in food packaging applications. [22]

Nonwovens

Advantages in properties over other polymers include softness, handfeel, drapability, air permeability, stretch-recovery, printability, and heat fusability.

Engineering components

As with many other plastics, the addition of glass fiber reinforcement significantly increases tensile strength, modulus of elasticity, bending strength and modulus, impact strength, and heat deflection temperature in molded parts of 3GT. In addition

to glass fibers, other natural fibers can be incorporated in 3GT to take advantage of lower process temperature and to increase total bio-derived materials content in the final product.

Creating the Future

Direction and Vision

The development and commercialization of bio-based Sorona[®] polymer by DuPont is consistent with the sustainable growth objectives of the company in its third century and beyond, through

- Knowledge Intensity: increased knowledge component of our products
- Productivity Improvement: reduced capital, environmental footprint, energy/resource usage, waste, emissions, and social cost
- Integrated Science: historically, chemistry and physics; in the future, chemistry, biology, physics, and information science

Realization

Carrying these principles forward, by DuPont and others, will lead to

- Greater use of renewable biological resources such as crop vegetation
- Further development of biological manufacturing processes, e.g., fermentation, (“biorefinery”) for intermediates and end products
- Products with integrated information technology, such as “smart” garments that automatically sense need for insulation vs. cooling/ventilation and respond accordingly

Conclusion

Sorona[®] polymer provides functionality and attributes different from that of other polymeric materials in the market today. To be successful, in addition to being a bio-derived material, products also need to provide value and functionality. With Sorona[®] polymer, either alone or in conjunction with other polymers, a variety of functionalities can be developed. From the 3G based platform other similar polymers can also be developed to satisfy growing needs of society.

Sorona[®] polymer is an example of DuPont's commitment to providing value-added products to the marketplace consistent with its responsibility both to its stakeholders and to the environment.

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- 09 US Patent 6,284,930 Process for the production of 3-hydroxypropanol
- 10 US Patent 6,281,325 Preparation of Poly(trimethylene terephthalate)
- 11 WO 0158980 Continuous process for producing Poly(trimethylene terephthalate)
- 12 US Patent 6,353,062 also EP 1254188 Continuous process for production of Poly(trimethylene terephthalate)
- 13 US 6,331,264 Low emission polymer compositions
- 14 US 6,325,945 Process of making a polyester or polyol
- 15 US 6,277,289, also WO 0102305; EP 1200358 Treatment of aqueous aldehyde waste streams
- 16 US 6,255,442 Esterification Process
- 17 US Patent 6,458,455 Poly(trimethylene terephthalate) tetrachannel cross-section staple fiber
- 18 US Patent 6,383,632 Fine denier yarn from Poly(trimethylene terephthalate)
- 19 EP 1183409 Poly(trimethylene terephthalate) yarns
- 20 US Patent 6,287,688 Partially oriented Poly(trimethylene terephthalate) yarns
- 21 US Patent 6,333,106 Draw textured Poly(trimethylene terephthalate) yarn
- 22 US Patent Application 20020012807 A1 (1/31/2002) Low temperature heat

sealable polyester film and method for producing same
23 US Patent 6,458,304 Flash spinning process and solutions of polyester
24 US Patent 6,245,844 also EP 1114095 Nucleating agent for polyesters

Figure Captions

Figure 1. Molecular structure of 3GT
Figure 2. Molecular shape and crystal structure of 3GT
Figure 3. Comparison of tensile characteristics
Figure 4. Comparison of stretch recovery characteristics
Figure 5. Comparison of dyeing characteristics
Figure 6. Product Life Cycle: Environmental Effects
Figure 7. Conversion of glucose to PDO in nature occurs in two steps
Figure 8. Pictorial representation of process: Corn to PDO
Figure 9. Pilot scale fermentation facility of Tate & Lyle, Decatur IL, USA
Figure 10. Purity Comparison of Bio and Chemical PDO
Figure 11. 3GT continuous polymerization process schematic
Figure 12. Refractive index chromatogram of production sample of Sorona® polymer
Figure 13. Viscosity characteristics of production sample of Sorona® polymer
Figure 14. DuPont product strategy for sustainable development and environmental responsibility

Tables

Table 1. Polymer and fiber properties
Table 2. Basic properties of 1,3-propanediol (PDO)
Table 3. Specific analytical properties of 1,3-propanediol (PDO) used for polymer manufacture
Table 4. Properties of continuously polymerized 3GT