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Elastomeric multi-filament as elastane alternative

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Summary

D2.7 examines the spinnability of the materials shortlisted in D2.3. As part of Task 2.3, these alternative materials to elastane are expected to contribute to develop closed-loop recyclable products, preferable as main components for sportwear applications, swimsuits.

Approval

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SCIRT.

SYSTEM CIRCULARITY & INNOVATIVE RECYCLING OF TEXTILES

Task 2.3: Primary materials

Deliverable D2.7: Elastomeric multi-filament as elastane alternative

Version N°1

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Disclaimer

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Keywords

Primary materials, sustainable materials, renewable materials, environmental performance, economic potential

Abbreviations and acronyms

Acronym	**Description**
CETI	European Center for Innovative Textiles
D	Deliverable
CAGR	Compound annual growth rate
Pa	Pascal
Psi	Pounds per square inch
°C	Celsius
L/D	Length/Diameter
TDS	Technical Data Sheet
V_r	Velocity rotation
T_r	Temperature rotation
TPE	Thermoplastic Elastomer
TPU	Thermoplastic Polyurethanes
TPE-E	Thermoplastic Copolyester Elastomer
TPE-A	Thermoplastic Copolyamide
PBAT	Polybutylene Adipate Terephthalate

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1 Introduction

The high demand for elastane fabrics across the world for manufacturing sportswear, socks, hosiery, swimwear and other clothing is expected to further grow the elastane fiber market. Looking forward, it is expected that the market reaches a value of US$ 16.87 Billion by 2030 exhibiting a CAGR of 7.31% during 2020-2030. However, manufacturing elastane fabrics involves the use of harmful chemicals, and if these chemicals are not disposed of properly, they could damage the environment. Moreover, elastane is not biodegradable and current recycling methods do not offer any real advantage to the recycling of fabrics containing elastane.

D2.3 presented a state of the art of new (preferably bio-based) polymers that can replace (fossil-based) elastane and can contribute to the development of closed-loop recyclable textile products. However, from an economic perspective, it was mentioned that the production of bio-based plastics is still much more expensive than fossil-based, mainly due to the low oil price, high investment costs of new infrastructure, and the current energy crisis.

If preferable bio-based solutions are needed, Mitsubishi, Lubrizol, BASF and Arkema were found to have solutions for alternative materials to elastane, as shown In Table below

Among worldwide material suppliers, BASF and Arkema were found to have bio-based solutions to replace elastane in Europe, which would limit transport and therefore carbon footprint.

Although D2.3 focused on alternative materials to elastane, the state of the art also included alternative solutions to elastane that are suitable for both thermo-mechanical and mechanical recycling processes. D2.3 showed that TPE-A and TPE-E based materials, such as PBAT and Pebax, have already been investigated and tested to deliver textile to textile solutions; PBAT staple fibers can be blended with cotton and wool to create textile products for the food industry and disposable protective clothing.

Overall, the state of the art conducted during D2.3 suggested that those materials provided by BASF and ARKEMA are preferable, since they are bio-based, local, and have been reported to work well for melt spinning applications and circular textile recycling solutions.

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Table 1 Shortlisted materials of D2.3

Supplier		Name	Category	€/kg	Melt spinning grade	Thermo-mechanical recycling	Mechanical recycling
Mitsubishi	JP	Tefabloc A	Bio-based	Not found	To be examined	YES	Not found
Mitsubishi	JP	Tefabloc R	Recycled materials	Not found	To be examined	YES	Not found
Lubrizol	US	Estane Eco	Bio-based	Not found	To be examined	YES	Not found
BASF	DE	Ecoflex F Blend	Bio-based	5	YES	YES	YES
Arkema	FR	Pebax 30R51	Bio-based	18	YES	YES	YES
Arkema	FR	Pebax 40R53	Bio-based	18	YES	YES	YES

2 Aims and Objectives

D2.7 examines the spinnability of the materials shortlisted in D2.3. As part of Task 2.3, these alternative materials to elastane are expected to contribute to develop closed-loop recyclable products, preferable as main components for sportwear applications, swimsuits.

3 Scope of research

To evaluate the spinnability of the materials shortlisted in D2.3 and prototype filaments whose properties must be similar to elastane or lycra, CETI has conducted an experimental program that includes 3 mayor steps. These are: 1- Rheometry, 2- Multifilament yarn prototyping, and 3- Monofilament yarn prototyping.

Rheometry was conducted to examine the suitability of the materials for use in melt spinning applications.

Multifilament yarn prototyping was performed to select the materials that best match the specifications of monofilament yarn prototyping. This study includes test of extrudability and spinning evaluations.

Since lycra or elastane in the clothing sector, especially in sportwear applications/swimsuits, is used as a knitted product, monofilament yarns must be prototyped to validate whether the materials might exhibit similar properties to lycra. Monofilament yarn prototyping is then the last validation step of CETI's experimental program.

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In this particular D2.7, monofilaments yarns were also designed on a lab scale spinning line located in a manufacturing plant in France, Chapter 7, with the aim of further study the suitability of the materials as alternative solutions to elastane.

4 Background and literature review

4.1 Rheology

The rheological properties of a polymer are studied using a capillary rheometer as shown in Figure 1.

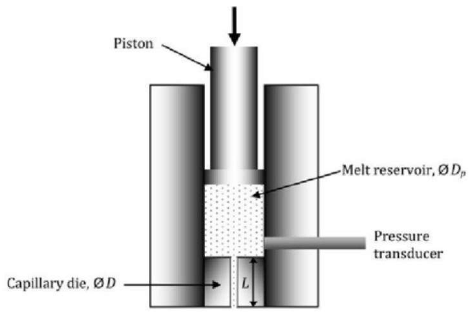

Figure 1: Capillary rheometer[1]

The measuring apparatus comprises a piston which subjects the polymer to the desired shear rates, a reservoir containing the liquid polymer to be studied, a pressure transducer to enable the viscosity of the polymer to be analyzed and a capillary die through which the material passes and undergoes the stress to be applied.

The polymer is pressurized and heated to pass through the capillaries of the spin pack. It is during the passage of the material through the spin pack that it will be subjected to the greatest shear rate. Therefore, the behavior of the polymer in this area must be predicted.

The viscosity of the polymer depends on two parameters: the temperature and the shear rate applied[2], as shown in the Carreau-Yasuda model below:

$$\eta\,(\dot{\gamma},T) = \frac{\eta_o\, f(T)}{[1+(r\dot{\gamma} f(T))^a]^{\frac{1-n}{a}}}$$

β: coefficient of temperature sensitivity, η_0: zero-shear viscosity, r: viscous relaxation time, n: power law index, a: width of the transition between zero-shear viscosity region and power law region

For a fixed shear rate, the viscosity only depends on the temperature, hence the importance of this parameter since the value of the shear rate is known.

The shear rate at the spin pack must be known beforehand and can be calculated using the following formula[2]:

$$\dot{\gamma} = \frac{\pi \times D \times N}{60 \times h}(s-1)$$

where D is the screw diameter in mm, N is the screw speed in rpm and h is the channel depth in mm.

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The rheometer will allow this shear rate to be reproduced to study the viscosity of the polymer.

In parallel, the rheometer will allow measurements to be taken at different temperatures in order to know the temperature at which the polymer will have the desired viscosity for melt spinning applications. In melt spinning applications, the polymers must have a viscosity between 50 and 100 Pa.s over a shear rate of $3000 s^{-1}$.The temperature must be increased when the viscosity is too high and inversely. This is shown in Figure below.

Moreover, the viscosity of the polymer must be sufficient for the polymer to flow through the capillary(s), but not exceed a certain limit beyond which the polymer is degraded and no longer has mechanical strength.

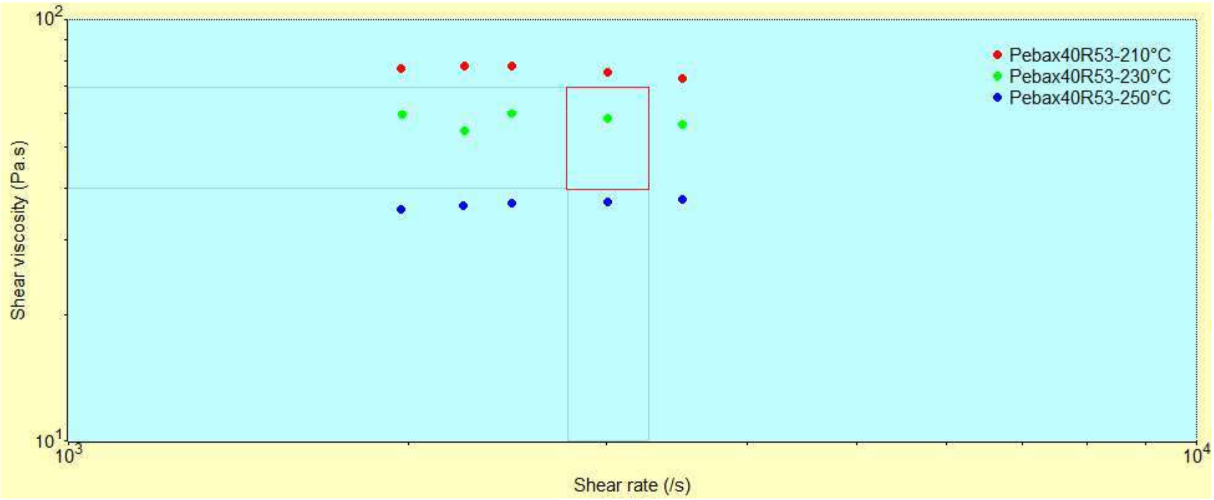

Figure 2 : Viscosity requirements for melt spinning applications

These studies on the rheological behavior of the materials will allow the temperature of the spin pack to be adjusted during the spinning process and therefore the temperature profiles to be inferred.

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4.2 Melt spinning: description

The schematic overview of a melt spinning process is illustrated below. The process involves 4 major stages and these are: 1 – extrusion, 2 – spinning, 3 – cooling and 4 - drawing and winding.

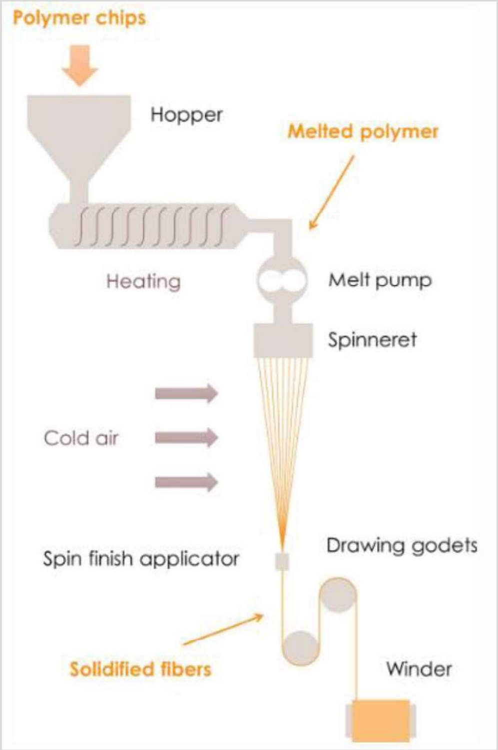

Figure 3 : Melt spinning line[3]

4.2.1 Extruder

Extrusion is a shaping process for thermoplastic polymers. The polymer is inserted in the form of pellets and its melting is carried out by heaters (external source) and internal heat from viscous energy dissipation via shearing. Extruders can have different characteristics such as: different screw profiles and diameters, different number of heating zones, extruder length, etc. All these characteristics will have an importance on the properties of the material after extrusion.

The main components of the extruder are the drive and feed systems, the screw, the barrel and the heating elements, the head and die assembly and the control system, as shown in Figure 4.

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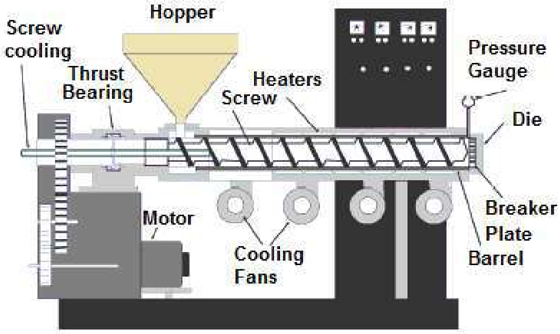

Figure 4 : Single screw extruder[4]

4.2.2 Extrusion screw

The screw of the extruder is a very important element in the extrusion process and its geometry should be adapted to the polymer so that it can be extruded in the best conditions.

The screw is driven by a motor in a heated cylinder sheath that can be adjusted to different temperatures. The sheath can be separated into 3 zones as shown in Figure 5, such as:

- Zone 1: Feed zone, feedstock is moved from hopper and preheated.
- Zone 2: Compression section, the polymer is transformed into a melt fluid and the material is compressed.
- Zone 3: Metering section, the melt is homogenized, and sufficient pressure is developed to pump it through the die opening

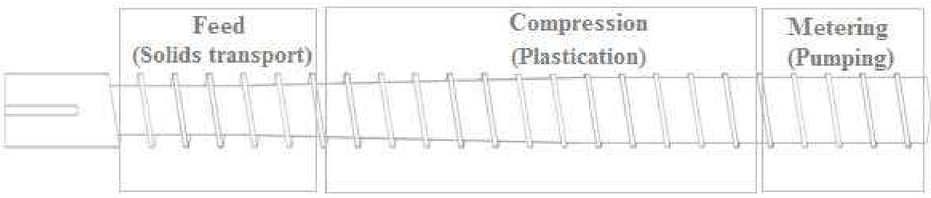

Figure 5 : Screw extruder design[5]

The material is exposed to high shear in zone 2 and this shear will greatly increase its temperature. The electrical energy supplied to the extruder will also be transformed into mechanical energy so as to rotate the screw, sending the polymer along the screw and increasing the pressure.

The internal friction will create heat and melt the polymer, and this is mainly responsible for the rise in temperature of the material. The residence time of the extrusion process is also important, and it should be long enough to allow the material to melt but short enough to avoid further degradation.

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The screw can have different profiles depending on the materials to be processed. The reference parameter to qualify a screw is its L/D ratio which represents the length of the barrel compared to the diameter of the screw[2], as presented in Figure 6. This ratio has an influence on the throughput. A higher L/D ratio have more melting and mixing capacity and can be run at higher rates.

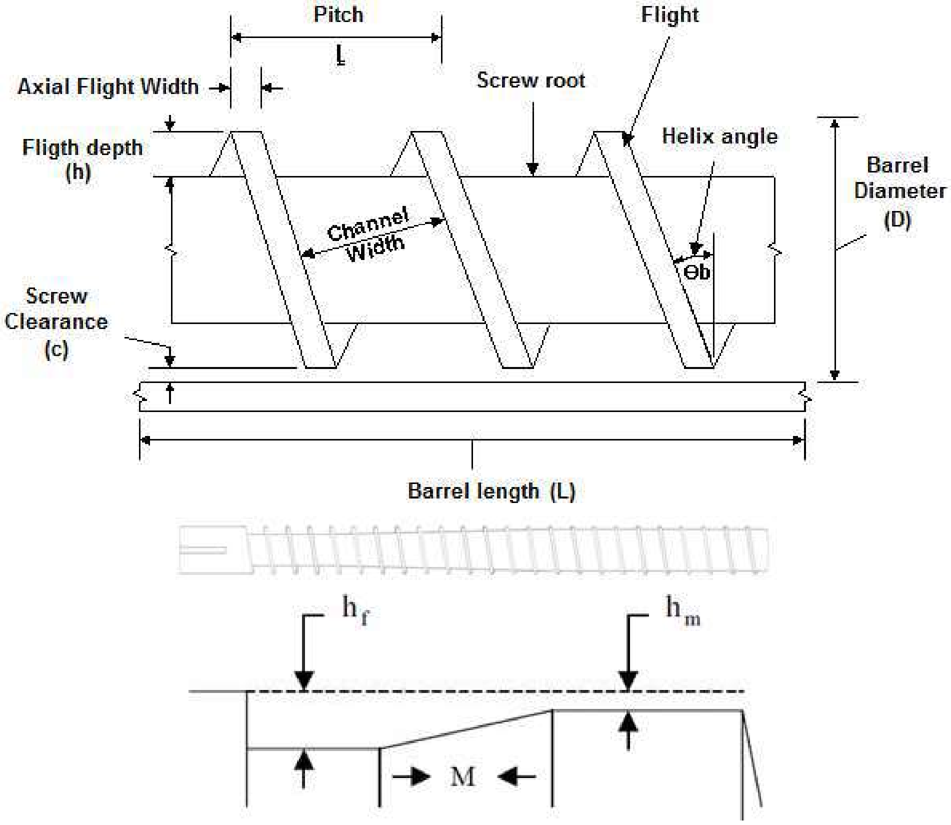

Figure 6 : The key dimensions of a conventional extruder screw[5]

Compression ratio and rate are important parameters for designing screw geometries since these affect the melting mechanism that occurs in the transition section of the screw.

Compression ratio is defined as the ratio between the depths in both the feed (h_f) and the metering section (h_m). Compression rate is used to measure the change in depth across the plastication section of the screw.

In the case of TPUs, the screws must have an L/D ratio of at least 24:1. The transition zone should be long and gradual, with a long metering zone up to half the total length of the screw. Due to their relatively high elastic recovery, TPU resins exhibit significant matrix swelling.

For regular engineering plastics, the value of the compression ratio is 3, and for TPUs is 1.

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4.2.3 Spinning

Melt-spinning is a widespread process that exclusively processes polymeric materials, and more particularly thermoplastic polymers that can be melted. This method of spinning is the most economical and is more environmentally friendly than solution dry spinning because it does not require solvent and because of the simplicity of the process, and moreover the speed of production of the filaments is 3 to 4 times faster.

The polymers are inserted in the form of pellets into the screw extruder. The number of different polymers that can make up the filaments varies according to the number of different extruders; for a 3-component spinning machine, 3 extruders are available to put from 1 to 3 different materials. The material will be heated to a temperature lower than the degradation temperature but higher than the melting temperature so as to obtain the polymer in liquid form without degradation.

A metering pump will keep the flow of molten polymer constant and move it from the extruder to the spinning head and then to the spin pack. The spin pack can be changed depending on the filaments requested. Indeed, it can be composed of one or more holes to respectively form mono or multi filaments, the size and shape of the holes can be very variable.

Once out of the spin pack, the polymer in the form of a filament will cool and solidify, either at room temperature, in cold air or in a water bath. These different methods and the associated temperatures depend on the polymer and type of filament; its shape and diameter, the properties to be obtained and the time spent cooling before being stretched.

The filament will then be able to be mechanically stretched by godets before being wound, which will allow the molecules to be aligned along the axis of the filament and influence the mechanical properties. The godets can take different temperatures to keep the polymer at the glass transition temperature[6].

The spin pack is a part that is located after the metering pump. It is through this part that the polymer must pass and take the desired shape. The spin pack is a removable element that consists of multiple distribution plates on top of a spinneret plate. The spinneret can have multiple holes with different cross-sections: round, hollow or trilobal, and different sizes. As explained in 4.1, the polymer will undergo a very high shear rate at this point, so the temperature of the spin pack should be adjusted according to the viscosity to be achieved.

The material must be cooled as soon as it leaves the spinneret, either by immersion in water or by contact with ambient or refrigerated air, in order to retain its shape.

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4.2.4 Quenching and Drawing

The structure and properties of polymeric fibers are determined by composition, molecular structure, crystallinity and molecular orientation. Crystallinity and orientation together determine the physical properties of fibers.

Quenching

Crystallinity is characterized by its percentage of crystalline area composing the polymer. These zones are due to an organization of the molecules which occurs preferentially between the glass transition temperature and the melting temperature. When the polymer is cooled, the chains move less and less and begin to order into crystallites, creating crystallization. In addition, stretching will bring the molecules closer to each other, increasing the interactions between them, again creating crystalline areas. The amorphous zones are between the crystallites. The crystallization of the polymer will depend on the cooling method and therefore on the cooling rate, but also on the stretching rate, bringing the molecules closer or less. The crystallinity will increase the tenacity of the fibers while the amorphous zones will increase the elasticity. When the cooling is slow, the crystallization has time to be optimal, whereas if the cooling is too fast, the components do not have time to crystallize.

There are different ways to cool a polymer, such as: cooling with ambient air, cooling in a cold-water bath and cooling with cold air jets.

Air cooling is the simplest way to crystallize a polymer, no apparatus is required. This method is used when the filament has a low denier, it can cool and crystallize before being stretched. When cooling by contact with ambient air is not sufficient and the filaments are also very thin, an air jet cooling device can be added, which is not available at CETI. For filaments with a higher denier, it is possible to use a cold-water bath and to regulate the water temperature for a better control of the mechanical properties of the filament produced. The choice of method depends on the type of filament produced and the desired mechanical properties.

Drawing

Molecular orientation is significant of the extension of macromolecules and structural units along the fiber axis. The orientation takes place at the level of drawing, the fibers lengthen, and the molecules are organized along the spinning axis. This parameter is mainly influenced by the speed of the stretching godets which will stretch the outgoing polymer more or less quickly. It is also influenced by the godet temperatures and the distance over which it is cooled because during drawing, the molecules will have more or less time to organize themselves in the direction of the spinning. The alignment of the molecules along the axis of the fiber will increase the mechanical strength of the fibers, in particular the tensile strength, but will decrease the elongation.

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4.3 Mechanical properties

To carry out mechanical measurements on the filaments, it is possible to use a dynamometer. This measuring instrument allows tensile and elasticity tests to be carried out automatically on the samples to be studied. The mechanical properties measured are: tenacity, maximum elongation and elastic recovery.

The instrument used at CETI is a Statimat, which consists of a test section made up of 2 clamps which hold the samples while the force and elongation measurements take place.

These tests ensure that the required filament properties are met. They are based on the final application of the filament.

Figure 7 : Statimat at CETI

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5 Experimental Equipment and Materials

5.1 Materials

The materials tested for melt spinning can be found in the "Alternatives" row of Table 2. According to technical data sheets (TDS), the polymers need to be dried in a dryer (see Annex 1 to 6).

Table 2 : Alternative materials to elastane

	Supplier	Grade	Polymer base	Biobased
References	Lycra Company	Lycra T275B	PU	No
		Lycra T275L	PU	No
Alternatives	Basf	Ecoflex F Blend C1200	TPE-E: PBAT	No
	Arkema	Pebax 30R51	TPE-A	Yes
		Pebax 40R53	TPE-A	Yes
	Mitsubishi	Tefabloc A06 75A W001	-	Yes
		Tefabloc R02 80A W001	-	Yes
	Lubrizol	Estane Eco 12T80E	TPU	Yes

5.2 Experimental Equipment

5.2.1 Rheology

The rheological behavior of all polymers was studied by a capillary rheometer. The instrument used was the RH2000 capillary rheometer from Malvern Instrument (see Figure 8).

The shear viscosity of each material was tested at 3 different temperatures in order to be able to compare the data and to gather information on the sensitivity of the polymers to temperature and shear rate. The rheometer was used according to the following parameters:

- Single bore measurements
- Shear rate: The rheological measurements were performed over a shear rate range of 2000 to 3500 s^{-1} in 5 steps. This range includes the shear rate experienced by the polymer as it passes through the spin pack, which is 3000s^{-1}.
- Pressure transducer: the sensitivity of the sensor is 10000Psi.
- Capillary die: this has a diameter of 0.5mm.
- Residence time: 8min so polymers can melt without degradation. This is also the residence time measured of the spinning process.

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Figure 8 : Capillary rheometer at CETI

5.2.2 Melt spinning

Spinning tests were carried out with the materials presented in Table 2 above.

To examine the extrudability and spinnability of the materials, tests were carried out with a multifilament spinneret. In total, 36 filaments were produced. A second trial campaign was also carried out to produce monofilaments, a single filament, Here, the specifications measured on these filaments were compared to reference filaments: Lycra T275B and Lycra T275L (see Annex). When lycra is used for swimsuit applications, the count of the filaments must range from 44 to 135dtex, and their mechanical properties are: elongation at break ±400%, tenacity: 0.9.

The specifications of the melt spinning line are shown below.

Figure 9 : Melt spinning line at CETI

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Table 3 : Specifications of the melt spinning line at CETI

Technical data	
Throughput	Up to 2 kg/h/circuit
Temperature	From 140 to 450°C
Extruders	19 mm (3/4”), L/D ratio : 30:1 - 3 heating zones
Melt pumps	A - 0,3 cc/rev - 10-90 rpm B - 0,6 cc/rev - 10-90 rpm C - 0,6 cc/rev - 10-90 rpm
Quench chamber	1,2m length, variable flow rate
Drawing godets	3 rolls Maximum speed: 2500 m/min Maximum temperature: 180°C
Speed – Winder	Up to 2500 m/min

Extrusion

The melt spinning machine located in CETI is a three-component spinning line, with three extruders available, as shown in Figure below. The trials were conducted with one component or extruder. For these particular studies, extruders C and A were chosen.

- *Extruder C* is preferred for multifilament production at high throughputs (0.6cc/rev). Trials were conducted with a flow rate set at 0.55 gr/hole/min, and the velocity of the pump was 30 rpm.
- *Extruder A* was used for monofilament production at low throughputs (0.3cc/rev). Here, trials were conducted with a flow rate set at 2gr/hole/min, and pump speed ranged from 6 to 7.75 rpm, depending on the material density,

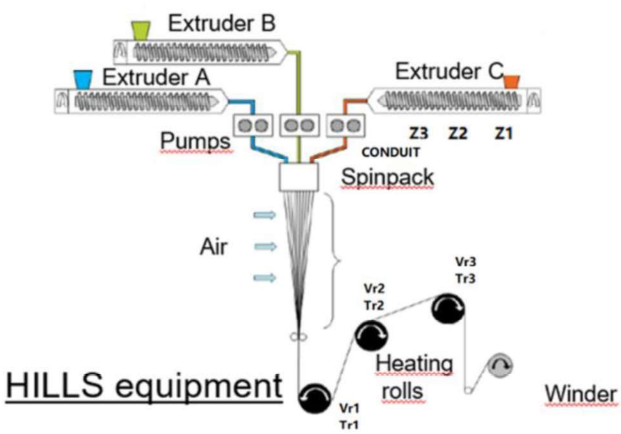

Figure 10 : Schematic diagram of the melt spinning line located at CETI

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Spin pack and temperature profiles

For multifilament and monofilament extrusion two spinpacks were used: 36 holes whose diameter is Ø 0.6mm, and 1 hole with a diameter of, Ø=0.9mm. Both multifilament and monofilament fibers had round cross section as shown in Figure below.

For extruder C and A, temperature profiles are defined as:

- Z1: zone 1 of the extruder which is the feed zone
- Z2: zone 2 of the extruder which is the compression section
- Z3: zone 3 of the extruder which is the metering section
- Pump
- Conduit
- Spin pack head

The temperature profile must gradually increase to make the melt reach optimal temperature in Z3, so that the melt can be pumped through the spin pack.

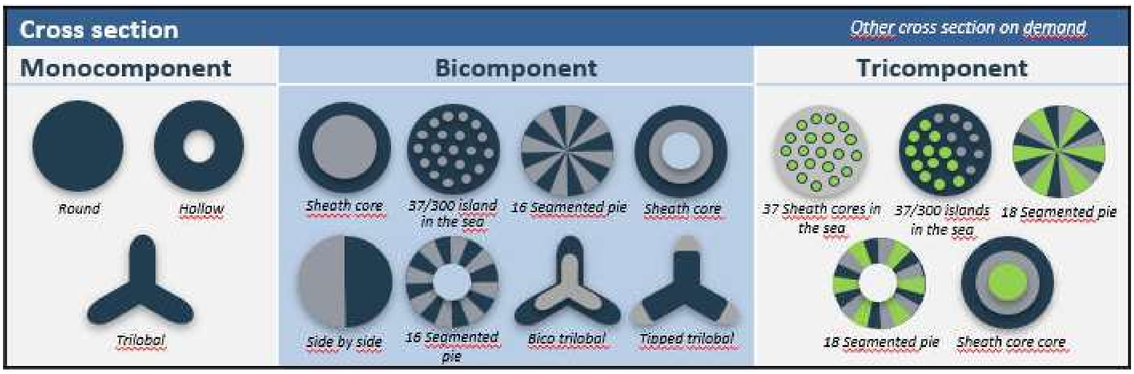

Figure 11 : Fiber cross sections

Quenching

Once out of the spinning machine, the filament changes from a molten to a solid state as it cools down and at this time it changes its crystal structure; recrystallize.

In multifilament spinning, the filaments are very thin and cooling in ambient air was found to be sufficient. This is air quenching at a room temperature of 25°C.

For monofilament spinning, the drying was done either by air quenching or by immersion in a water bath. For water bath cooling, tests were carried out in a manufacturing lab scale line in France.

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Drawing

The crystal structure will also be influenced by the stretch and heat provided by the drawing unit. In CETI, the melt spinning line has 3 godets (see Figure 10), and these can run at different rotation speeds, V_r , and temperatures, T_r .

In multifilament spinning, trials were conducted under two different scenarios: B1 and B2.

- Scenario B1

  BASF's Ecoflex FBlendC 1200 polymer is used as reference data. For this particular material, maximum godets speeds are very well known and these are:

  - Vr1= 330 m/min
  - Vr2= 500 m/min
  - Vr3= 510 m/min

  The drawing can be calculated as follow: Vr3/Vr1= 1.5. In this particular scenario, remaining materials are tested under the same conditions to provide a comparative analysis and determine whether a wider drawing process window can apply.

- Scenario B2

  This applies to those materials that can be drawn at higher godet speeds than BASF's Ecoflex FBlendC 1200. To find the maximum drawing for each polymer, the speed of the godet Vr2 is increased until the filaments are found to break. The speed of the godet Vr3 can be then calculated as Vr2+10.

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5.2.3 Mechanical properties

By providing the filament count, tenacity and elastic tests were conducted to determine the following mechanical properties: tenacity, elongation and the elastic recovery. Specifications of the tests can be found in Annex.

Figure 12 : Monofilament test: sample approaching its maximum elongation

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6 Results and discussion

In this section, the overall experimental approach conducted in D2.7 is discussed. The entire experimental program is depicted below. First, the rheological data of the selected materials will be discussed. In 6.2.1, the extrudability of the materials is presented and the results are carefully discussed for multifilament yarns. Here, two materials were found to not exhibit extrudability; these are Tefabloc A and Tefabloc R. Moreover, Ecoflex Fblend was found to exhibit a narrower drawing process window and was no longer further tested. By the experiments conducted in section 6.2.2, Estane Eco was found to be non-spinnable for monofilament applications, so Pebax 30R51 and Pebax 40R53 were chosen to finally perform a thorough analysis of their spinnability performance in both air and water cooling.

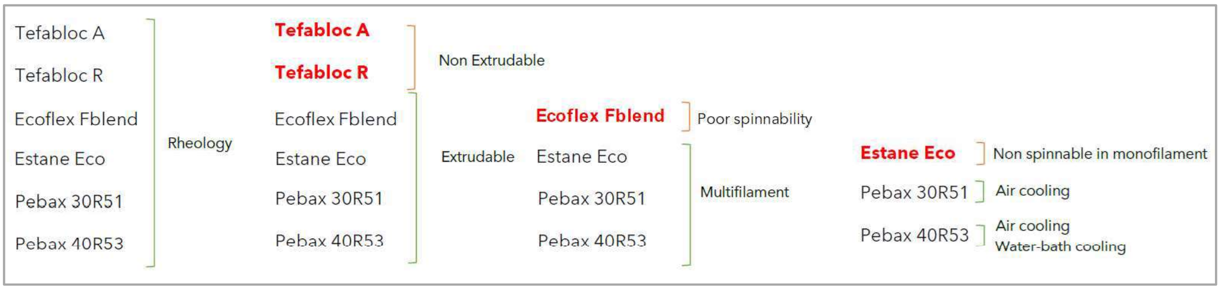

Figure 13 : Overall experimental program approach

6.1 Rheological results

- Lubrizol Estane Eco 12T80E

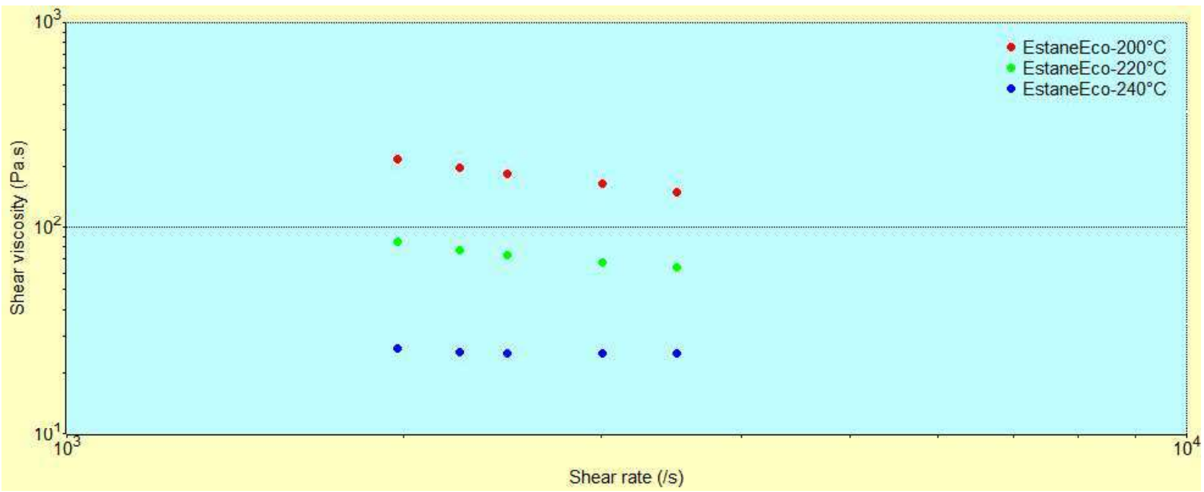

Figure 14 : Rheological analysis for Estane Eco 12T80E

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The material seems to be spinnable at a temperature around 220°C, since viscosity ranges from 50 and 100 Pa.s over a shear rate of 3000s^{-1}.

- Arkema Pebax 30R51

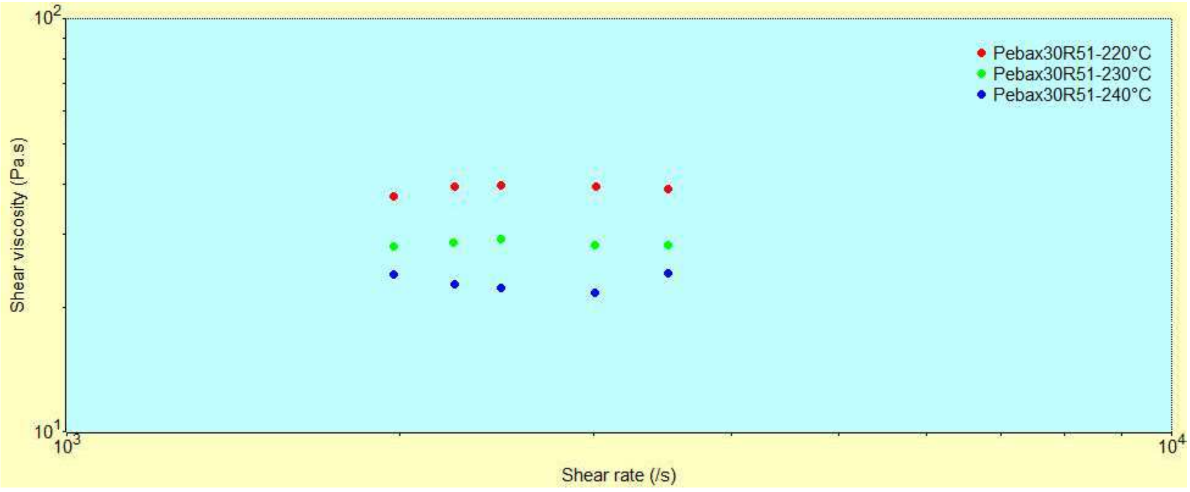

Figure 15 : Rheological analysis for Pebax 30R51

The material seems to be spinnable at a temperature around 220°C, since viscosity ranges from 50 and 100 Pa.s over a shear rate of 3000s^{-1}.

- Arkema Pebax 40R53

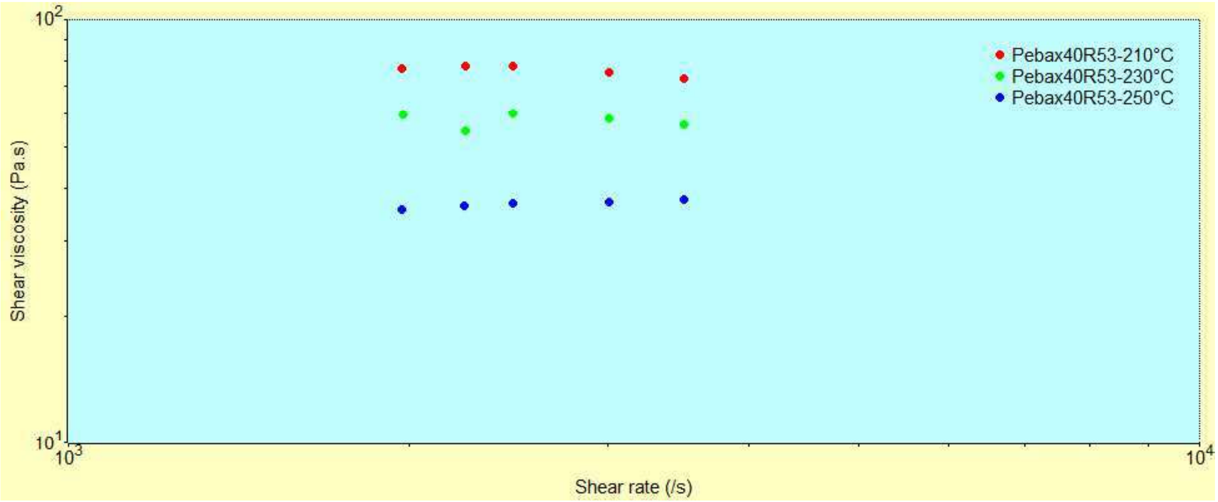

Figure 16 : Rheological analysis for Pebax 40R53

The material seems to be spinnable at a temperature around 230°C, since viscosity ranges from 50 and 100 Pa.s over a shear rate of 3000s^{-1}.

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

- MCPP Tefabloc TG SI A06 75AW001

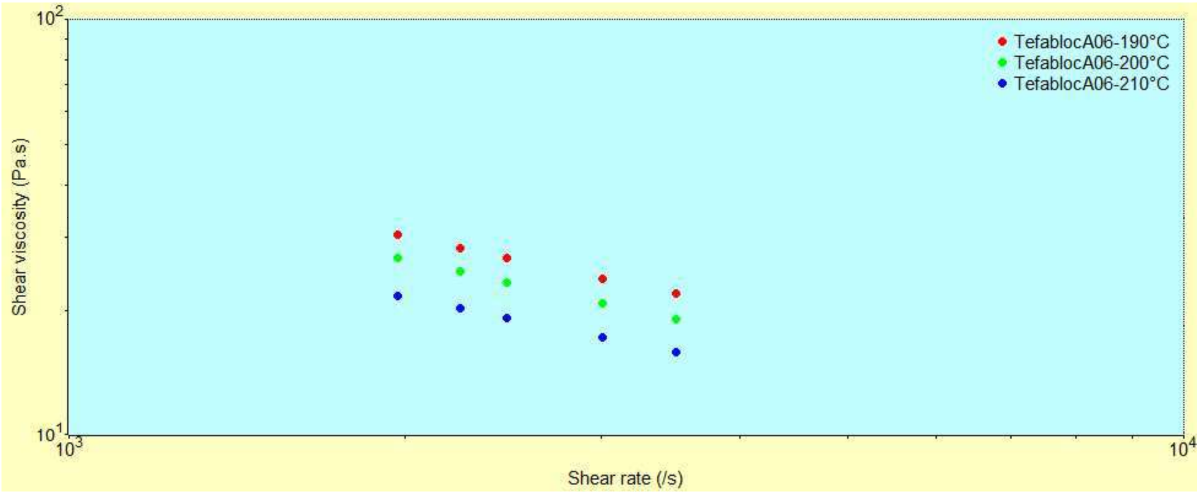

Figure 17 : Rheological analysis for Tefabloc TG SI A06 75AW001

The material seems to be spinnable at a temperature around 190 - 200°C, since viscosity ranges from 50 and 100 Pa.s over a shear rate of 3000s^{-1}.

- MCPP Tefabloc TG SI R02 80AW001

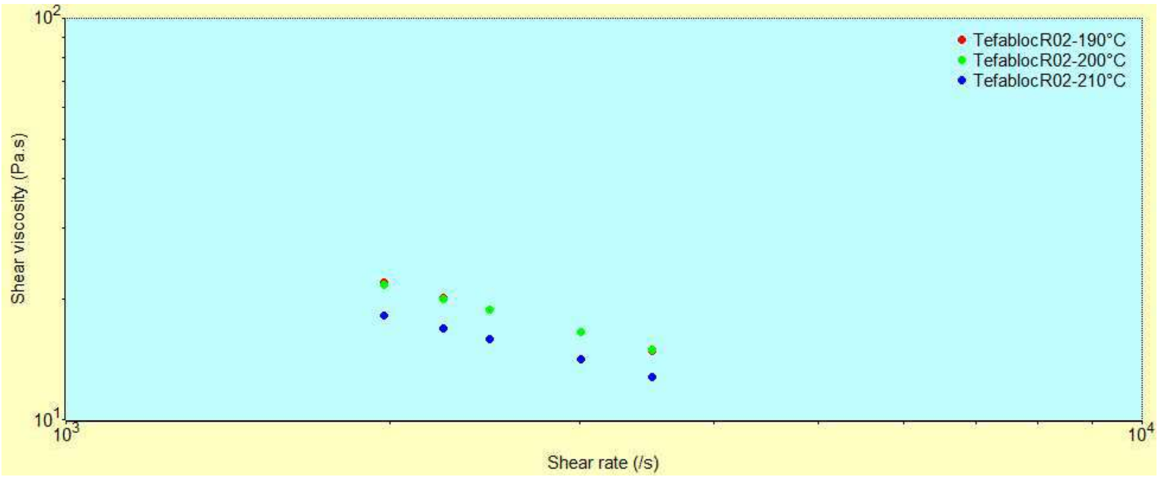

Figure 18 : Rheological analysis for Tefabloc TG SI R02 80W001

The material seems to be spinnable at a temperature around 190 - 200°C, since viscosity ranges from 50 and 100 Pa.s over a shear rate of 3000s^{-1}.

- BASF Ecoflex FBlendC1200

The rheological properties of this material are already known in CETI and are not presented in this report. The polymer is well known to be spinnable at 240°C.

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

Summary:

In this chapter it is shown that all polymers seem to be suitable for melt spinning applications when temperature is carefully studied. These results are of vital importance to stablish the extrusion temperature profile and the spin pack temperature that will best work for subsequent spinning trials, whose results are carefully discussed in section 6.2.

6.2 Melt spinning results

6.2.1 Multifilament yarns

Temperature profiles

Extruder C was used for multifilament extrusion and validation of extrudability. The spin pack used had 36 holes of 0.6mm diameter, and L/D = 4.

The temperatures are set up according to the information gathered from rheology and these can be found in Table below.

Table 4 : Barrel temperature profiles

Barrel temperature profile	Z_1 (°C)	Z_2 (°C)	Z_3 (°C)	Pump (°C)	Conduit (°C)	Spin-pack head (°C)	gr/hole/min	Pump (rpm)
Ecoflex FBlendC 1200	190	210	220	230	230	240	0.55	30
Pebax 30R51	190	200	210	220	220	230	0.55	30
Pebax 40R53	190	200	210	220	220	230	0.55	30
Tefabloc TG SI A06 75AW001	190	195	200	200	200	215	0.55	30
Tefabloc TG SI R02 80AW001	190	195	200	200	200	215	0.55	30
Estane Eco 12T80E	195	200	210	220	220	225	0.55	30

Trials were conducted to determine which polymers were melt-extrudable and melt-spinnable in multifilament.

- Ecoflex FBlendC 1200, Pebax 30R51, Pebax 40R53 and Estane Eco were found to be extrudable. This is though to result from the pressure measured at the pump, which exhibited very little fluctuations, leading to a constant flow of material out of the extruder.
- Ecoflex FBlendC 1200, Pebax 30R51, Pebax 40R53 and Estane Eco were also found to be melt-spinnable. These materials showed an elastic character but did not present any difficulties when passing through the spin pack

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

- Tefabloc TG SI A06 75AW001 and Tefabloc TG SI R02 80AW001 were found to be non-extrudable and therefore non-melt spinnable. The extrusion pressure was found to exhibit large fluctuations. This is thought to result from the design of the screw. As shown in Chapter 4.2.2, the extrusion screw has a compression ratio of 3, which might not match the requirements of these particular materials. With these two materials, the spinning test failed, and filaments broke at the exit of the spinneret. Moreover, the results are in agreement with Table 2, at which the chemistry of both materials is not disclosed and their polymer-based nature cannot be confirmed to be TPE or TPU based.

Drawing

This section presents the materials that can be used for multifilament applications. Trials were conducted under two different scenarios:

- B1: Ecoflex FBlendC 1200 maximum godet speeds. This can be found in Table 5
- B2: maximum drawing process window

Table 5 : Ecoflex FBlendC 1200

Sample	Ø Spinneret (mm)	Vr1 (m/min)	Vr2 (m/min)	Vr3 (m/min)	Drawing (Vr3/Vr1)	Tr1 (°C)	Tr2 (°C)	Tr3 (°C)	Spinning Performance
Ecoflex FBlendC 1200	0.6	330	500	510	1.5	n/a	n/a	n/a	Good

Table 6 : B2: maximum drawing process window

Sample	Ø Spinneret (mm)	Vr1 (m/min)	Vr2 (m/min)	Vr3 (m/min)	Drawing (Vr3/Vr1)	Tr1 (°C)	Tr2 (°C)	Tr3 (°C)	Spinning Performance
Pebax 30R51	0.6	330	1650	1660	5	n/a	n/a	n/a	Good
Pebax 40R53	0.6	330	860	870	2.6	n/a	n/a	n/a	Good
Estane Eco	0.6	330	825	835	2.5	n/a	n/a	n/a	Good

Summary:

Pebax 30R51, Pebax 40R53 and Estane Eco were found to exhibit a wider drawing process window than Ecoflex FBlendC 1200, as shown in Table 6. As a result, the monofilament performance of Ecoflex FblendC 1200 was no longer examined.

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

6.2.2 Monofilament yarns

To examine whether the materials can be finally used to replace elastane/lycra for use in the clothing sector, swimwear applications, monofilament yarns must be produced with a yarn size ranging from 44dtex to 135dtex. At 44 dtex, elongation at break is measured at ≥400%. Trials were then conducted to examine the drawing process window for each material so that different yarn sizes can be designed.

Temperature profiles

Trials were conducted with extruder A, and the spin pack installed on the spinning line had a single hole of 0.9mm in diameter. The flow rate was set at 2gr/hole/min.

Temperatures profiles were defined according to rheometry, and these can be found in Table 7.

Table 7 : Temperature profiles in multifilament

Barrel temperature profile	Z_1 (°C)	Z_2 (°C)	Z_3 (°C)	Pump (°C)	Conduit (°C)	Spin-pack head (°C)	gr/hole/min	Pump (rpm)
Pebax 30R51	195	200	210	220	220	225	2	6,6
Pebax 40R53	190	200	210	220	220	215	2	6,6
Estane Eco 12T80E	195	200	210	220	220	225	2	6

- For Estane Eco, monofilament yarns failed because the filament was too sticky and could not be wrapped onto the godets.
- For Pebax 30R51 and Pebax 40R53, air quenching cooling was found to be sufficient to cool down the monofilament.

Drawing

Drawing settings for Pebax 30R51 are presented below.

Table 8 : Drawing stand settings for Pebax30R51

Sample	Ø Spinneret (mm)	Vr1 (m/min)	Vr2 (m/min)	Vr3 (m/min)	Winder (m/min)	Drawing (winder/Vr1)	Tr1 (°C)	Tr2 (°C)	Tr3 (°C)	Spinneret performance
I	0.9	110	120	130	1075	9.8	n/a	n/a	n/a	Limit
II	0.9	110	120	130	1000	9.1	n/a	n/a	n/a	Good
III	0.9	110	120	130	400	3.6	n/a	n/a	n/a	Good
IV	0.9	110	120	130	270	2.5	n/a	n/a	n/a	Good

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

- This polymer can be spun at different winder speeds. Here, it must be noticed that drawing was shifted and took place between the final godet and the winder
- The drawing window was found to range between 2.5 and 9.8.

Drawing settings for Pebax 40R53 are presented below.

Table 9 : Drawing settings for Pebax40R53

Sample	Ø Spinneret (mm)	Vr1 (m/min)	Vr2 (m/min)	Vr3 (m/min)	Winder (m/min)	Drawing (winder/Vr1)	Tr1 (°C)	Tr2 (°C)	Tr3 (°C)	Spinneret performance
I	0.9	110	120	130	1000	9.1	n/a	n/a	n/a	Limit
II	0.9	110	120	130	660	6.0	n/a	n/a	n/a	Good
III	0.9	110	120	130	400	3.6	n/a	n/a	n/a	Good
IV	0.9	110	120	130	270	2.5	n/a	n/a	n/a	Good

- Similar to Pebax 30R51, drawing was shifted and took place between the final godet and the winder
  The drawing window was found to range between 2.5 and 9.1.

Summary:

As the aim was to obtain very fine monofilaments of 44dtex, the limits of the spinning machine were reached. Indeed, it is impossible to have a flow rate lower than 2gr/min and a pump speed lower than 8rpm, which made the drawing to be shifted to reach the desired fineness.

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

6.3 Mechanical properties

The filament count must be determined in a first place to set the Statimat, and determine some mechanical properties such as: tenacity, elongation and the elastic recovery.

6.3.1 Multi filament yarns

The bobbins named B1 represent the bobbins made with the maximum speeds of the reference material, Ecoflex. The B2 bobbins represent the bobbins made with the maximum godet speeds for each multi-filament spinnable polymer.

Table 10 : Mechanical analysis in multi filament

		Ecoflex	**Pebax30R51**		**Pebax40R53**		**Lubrizol Estane Eco**	
		B1	**B1**	**B2**	**B1**	**B2**	**B1**	**B2**
Yarn count	Dtex	274	400	50.6	370	221	339	234
	Elongation %	37.2	350	127	178	88	161	68
	Tenacity cN/dtex	1.39	0.73	6.04	1.46	2.65	0.92	1.31
	Elastic recovery	-	-	-	-	-	-	-

The elastic recovery could not be measured because the modulus was not functional on the CETI measuring instrument.

From Table 10, it can be noticed that Ecoflex compares less favorable than Pebax 30R51, Pebax 4R53, and Estane Eco in terms of elongation at B2 so these results confirm why this particular material was no longer examined for monofilament applications.

6.3.2 Monofilament yarns

The mechanical results of the monofilament yarns are presented in Table below, when air quenching was used.

Table 11 : Mechanical analysis in mono filament

		Arkema Pebax30R51				**Arkema Pebax40R53**			
Yarn count	Dtex	48	60	105	135	45	60	100	135
	Elongation %	254	263	>500	>500	160	310	520	>500
	Tenacity cN/dtex	1.26	0.95	-	-	2.50	1.90	3.10	-
	Elastic recovery	-	-			-			-

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

To provide a comparative analysis, elongation measured on Pebax 30R51 and Pebax 40R53 is displayed in more detail in Figure 19. For lycra, elongation reaches 400% for a count of 44dtex. A dotted line has been then added to the graph to provide reference data.

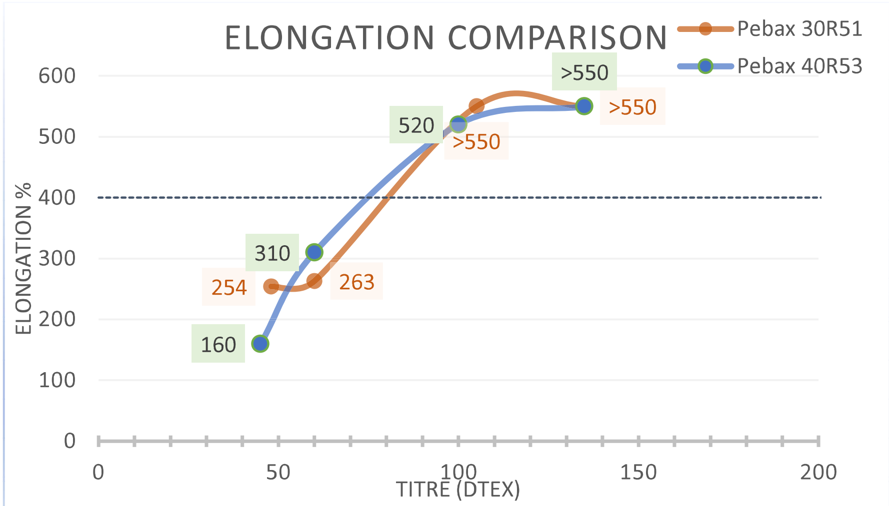

Figure 19: Elongation comparison in mono filament

The elongation values depend on the count of the monofilament, the higher the count, the higher the elongation. Elongation becomes sufficient at around 80dtex for Pebax 30R51 and Pebax 40R53.

Summary:

In CETI's line, diameter of the spinneret and flow rate were found to be limited processing factors, and drawing at a larger extension was needed to obtain fine count monofilaments.

This tended to drastically drop the elongation measured on the samples.

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7 Monofilament yarns: manufacturing plant

Additional monofilament trials were also conducted on a different lab scale manufacturing melt spinning line located in France to produce yarns whose specifications can be closer to lycra ones. The trials were conducted with 2 materials: Pebax 30R51 and Pebax 40R53.

Among these two materials, Pebax 40R53 was found to provide superior spinning performance than Pebax 30R51 on this particular line.

A schematic diagram of the equipment is presented in Figure 20.

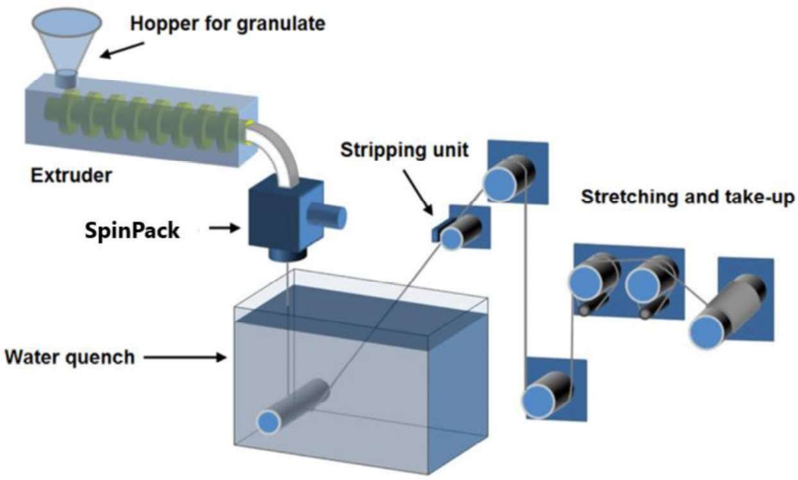

Figure 20 : Manufacturing lab scale spinning line

Compared to the process conducted in CETI with Pebax, the diameter of the spinneret was lower, Ø = 0.5mm. The line was then expected to produce low count filaments with limited drawing. Moreover, cooling took place in a water bath. Compared to air quenching, the line was also expected to produce fine count filaments with much higher elongation.

The trials are summarized in Table below. The material used was Pebax 40R53.

Table 12 : Test parameters for Pebax 40R53

Sample	Ø Spinneret (mm)	Vr1 (m/min)	Vr2 (m/min)	Winder (m/min)	Titer (dtex)
1	0.5	130	130	130	122
2	0.5	130	130	130	66
3	0.5	130	130	130	135
4	0.5	130	130	130	135

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

Table 13 : Temperature profiles for Pebax 40R53

Sample	Z_1 (°C)	Z_2 (°C)	Z_3 (°C)	Pipe (°C)	Spin-pack head (°C)	Power extruder (%)	Height spinpack-water (cm)	Water temperature (°C)
1	170	190	220	220	215	6	34	11
2	170	190	220	220	215	4	34	11
3	170	190	220	220	215	7	34	11
4	170	190	220	220	215	7	12.5	11

The design of experiments was:

Sample 1: Parameters were adjusted to achieve a titre of 122 dtex.

Sample 2: Compared to the previous trial, the titre is reduced to reach 66 dtex.

Sample 3: Upper limit titre (4.2.2) is achieved during these trials at 135 dtex.

Sample 4: The height between the bath and the die is minimized to compare filaments with the same titre range but with faster cooling.

Mechanical properties

Compared to Pebax 40R53 with air quenching, elongation measured on each sample was found to be ≥ 450%, which is thought to result from water cooling. For these particular monofilament yarns, elastic recovery was also measured in CETI. The results are summarized in Figure 21 below.

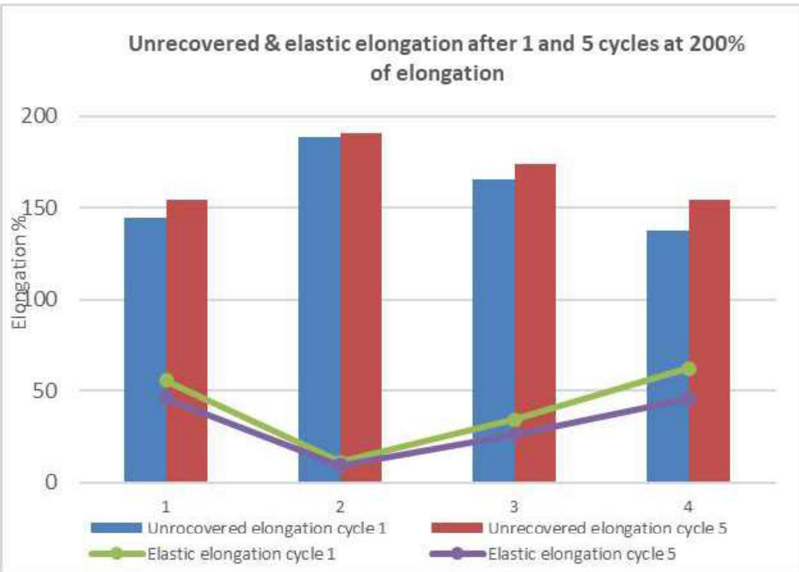

Figure 21 : Mechanical properties for Pebax 40R53 : elastic elongation

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

From Figure 21, when sample 1 compares to sample 2, it can be noticed that elastic elongation between cycle 1 and 5 was highly affected by the size/count of the filament. When sample 3 compares to sample 4, elastic elongation increased by decreasing the height or gap between the spinneret and the water bath, which is thought to result from faster cooling that tends to increase the amorphous nature of the filament.

Unfortunately, and compared to lycra, at 200% of elongation unrecovered elongation is expected to be 0 for pure elasticity. From Figure 21, unrecovered elongation measured for all samples was found to be too poor, especially those measured on cycles 5.

Summary:

Compared to Pebax40R53 with air quenching, water cooling was found to compare more favorable. When the diameter of the spinneret was lower, Ø=0.5, drawing did not apply drastically to produce fine filament and elongation measured on the samples exceed lycra specification, E≥400%. However, elastic elongation and unrecovered elongation were found to be too poor when these compare to lycra.

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

8 Conclusions

This study examined the performance of the alternatives materials shortlisted in D2.3, which should retain the exceptional properties of lycra whilst having an environmental advantage, i.e. be recyclable, compostable or biodegradable and/or be synthesized from biosourced or recycled raw materials. Moreover, the materials must be available on the market and preferably in Europe to limit their transport and therefore their carbon footprint.

In total, 6 materials were evaluated. These are: 1 – Tefabloc A and Tefabloc R from Mitsubichi, JP, 2- Estane Eco from Lubrizol, US, 3- Ecoflex F Blend from BASF, DE, and 4 – Pebax 30R51 and 40R53 from Arkema, FR.

The spinnability evaluation of the materials was conducted by a 3- step process that included: 1- rheometry, 2- multifilament yarn prototyping, and 3 – monofilament yarn prototyping. The goal was the design of a yarn whose elongation at break is higher than 450% for a yarn count of 45 dtex, and elastic deformation is completed reversible under 200% of elongation.

Through the thorough analysis presented in this D2.7, it can be concluded that lab scale spinning lines are not very well equipped and designed to perform a fine spinnability evaluation of these particular materials. Results shown, however, that the materials developed by Arkema showed superior spinning performance and can be used to design yarns whose size and elongation properties are closer to lycra. With Pebax 40R53, an elongation higher than 450% was measured for a yarn count of 66 dtex. Unfortunately, elastic deformation was found to be very poor compared to lycra.

The findings from this D2.7 agree with recent publications given at the Dornbirn Global Fiber Congress in Austria, where researchers from the Institute of Textile Technology at RWTH Aachen were presenting a novel melt spinning process which can produce new CO_2 based TPU materials whose performance should be superior to those measured on the materials examined in this work. In fact, this group is now partnering with Covestro to implement an industrial scale production process for making stretch fibers.

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

Results suggest that to come up with answers immediately to this topic, the most efficient solution is the partial replacement of lycra on knitted products to lower its content and design a product whose label is greener, improving its suitability for textile recycling. Although CETI has proven success in medical applications, mask earloops, this composite thread must be validated for the market applications needed in SCIRT.

Lastly, knitted samples with engineered patterns might be fabricated with the materials from ARKEMA for imparting improved elasticity.

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This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

Annex

Annex 1

CAMPUS® fiche technique

Pebax® Rnew® 30R51 SA 01 - TPA
ARKEMA

Informations produit

Polyether block **Pebax® Rnew® 30R51 SA 01 resin** is a thermoplastic elastomer made of flexible polyether and rigid polyamide based on renewable resources.
Pebax® Rnew® 30R51 SA 01 resin is an inherently antistatic polymer and can be dry blended or compounded with a polymer matrix to lower the surface resistivity of the final part. This grade is particularly recommended for PMMA matrices. This hydrophilic grade when extruded into either a thin film or laminated on to a substrate offers excellent permeability to moisture vapor while remaining waterproof.

The percentage of renewable carbon according to ASTM D6866 is **41%.**

Refractive index according to an internal method is 1.49.

Main applications:

- Permanent antistatic additive for PMMA matrices
- Breathable membranes
- Note: this grade is not recommended by Arkema for usage in medical applications

Packaging:
This grade is delivered dried in sealed packaging (25 kg bags) ready to be processed.

Shelf Life:
Two years from the delivery. For any use above this limit, please refer to our technical services.

Propriétés mécaniques	**sec / cond**	**Unité**	**Norme du test**
Module en traction	**- / 59**	MPa	ISO 527-1/-2
Contrainte pr. une déform. de 50%	**- / 7**	MPa	ISO 527-1/-2
Contrainte à la rupture TPE	**16 / ***	MPa	ISO 527-1/-2
Déformation à la rupture	**- / >50**	%	ISO 527-1/-2
Déformation à la rupture TPE	**>300 / ***	%	ISO 527-1/-2
Dureté Shore D, 15s	**30 / ***	-	ISO 7619-1
Propriétés thermiques	**sec / cond**	**Unité**	**Norme du test**
Température de fusion, 10°C/min	**150 / ***	°C	ISO 11357-1/-3
Propriétés électriques	**sec / cond**	**Unité**	**Norme du test**
Résistivité transversale	**1E8 / 1E8**	Ohm*m	IEC 62631-3-1
Résistivité superficielle	*** / 1E8**	Ohm	IEC 62631-3-2
Propriétés diverses	**sec / cond**	**Unité**	**Norme du test**
Absorption d'eau	**20 / ***	%	Sim. to ISO 62
Absorption d'humidité	**2.5 / ***	%	Sim. to ISO 62
Masse volumique	**1010 / -**	kg/m³	ISO 1183

Caractéristiques

Transformation
Moulage par injection, Extrusion (autres)

Propriétés spéciales
Antistatique, Stabilisé à la chaleur

Conditionnement
Granulés

Disponibilité régionale
Amérique du nord, Europe, Asia Pacific, South and Central America, Proche-Orient/Afrique

Autres informations

Moulages par injection

Last update: 2021-02-04 Source: https://www.campusplastics.com Page: 1/2

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

Pebax® Rnew® 30R51 SA 01 - TPA
ARKEMA

Processing conditions:
- Typical melt temperature (Min / Recommended / Max): 200°C / 240°C / 270°C.
- Typical mold temperature: 25–60°C.
- Drying time and temperature (only necessary for bags opened for more than two hours): 4-6 hours at 65-75°C.

Extrusion (autres)

Processing conditions:
- Typical melt temperature (Min / Recommended / Max): 210°C / 220°C / 230°C.
- Drying time and temperature (only necessary for bags opened for more than two hours): 4-6 hours at 65-75°C.

This database provides the main properties of technical thermoplastics marketed by ARKEMA.

The information contained in this document is based on trials carried out by our Research Centres and data selected from the literature, but shall in no event be held to constitute or imply any warranty, undertaking, express or implied commitment from our part.
Our format specifications define the limit of our commitment.
No liability whatsoever can be accepted by ARKEMA with regard to the handling processing or use of the product or products concerned which must in all cases be employed in accordance with all relevant laws and/or regulations in force in the country or countries concerned.

For information about the other products marketed by our company consult :
ARKEMA

Last update: 2021-02-04 Source: https://www.campusplastics.com Page: 2/2

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

Annex 2

CAMPUS® fiche technique

Pebax® Rnew® 40R53 SP 01 - TPA
ARKEMA

ARKEMA

Informations produit
Polyether block **Pebax® Rnew® 40R53 SP 01 resin** is a thermoplastic elastomer made of flexible polyether and rigid polyamide based on renewable resources. This SP grade has been developed to be heat and UV resistant.

The percentage of renewable carbon according to ASTM D 6866 is **44-48%.**

Main applications:

- Flexible injected parts

Packaging:
This grade is delivered dried in sealed packaging (25 kg bags) ready to be processed.

Shelf Life:
Two years from the delivery. For any use above this limit, please refer to our technical services.

Propriétés rhéologiques	**sec / cond**	**Unité**	**Norme du test**
Retrait au moulage, parallèle	**0.6 / ***	%	ISO 294-4, 2577
Retrait au moulage, perpendiculaire	**0.7 / ***	%	ISO 294-4, 2577
Propriétés mécaniques	**sec / cond**	**Unité**	**Norme du test**
Module en traction	**- / 75**	MPa	ISO 527-1/-2
Contrainte pr. une déform. de 50%	**- / 8.2**	MPa	ISO 527-1/-2
Contrainte à la rupture TPE	**45 / ***	MPa	ISO 527-1/-2
Déformation à la rupture	**- / >50**	%	ISO 527-1/-2
Déformation à la rupture TPE	**>300 / ***	%	ISO 527-1/-2
Résistance au choc Charpy, +23°C	**N / N**	kJ/m²	ISO 179/1eU
Résistance au choc Charpy, -30°C	**N / N**	kJ/m²	ISO 179/1eU
Résistance au choc Charpy, +23°C	**N / N**	kJ/m²	ISO 179/1eA
Résistance au choc Charpy, -30°C	**N / N**	kJ/m²	ISO 179/1eA
Dureté Shore D, 15s	**39 / ***	-	ISO 7619-1
Propriétés thermiques	**sec / cond**	**Unité**	**Norme du test**
Température de fusion, 10°C/min	**148 / ***	°C	ISO 11357-1/-3
Température de ramolliss. Vicat, 50°C/h 50N	**121 / ***	°C	ISO 306
Propriétés diverses	**sec / cond**	**Unité**	**Norme du test**
Absorption d'eau	**1.4 / ***	%	Sim. to ISO 62
Absorption d'humidité	**0.5 / ***	%	Sim. to ISO 62
Masse volumique	**1030 / 1030**	kg/m³	ISO 1183

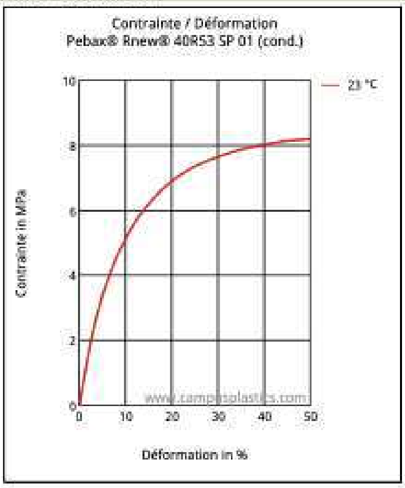
Last update: 2021-02-04 Source: https://www.campusplastics.com Page: 1/2

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

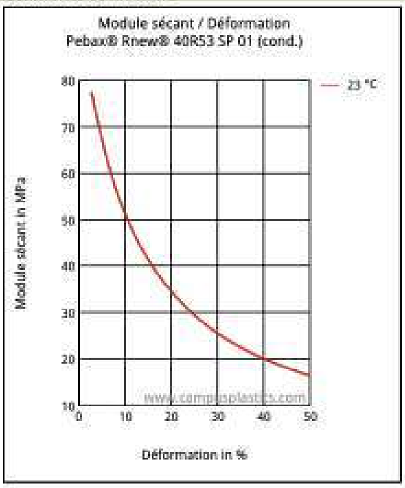
Pebax® Rnew® 40R53 SP 01 - TPA
ARKEMA

Fonctions

Contrainte / Déformation



Module sécant / Déformation



Caractéristiques

Transformation
Moulage par injection, Extrusion de films, Extrusion de profilés, Extrusion (autres), Moulage par transfert, Coulage, Thermoformage

Conditionnement
Granulés

Propriétés spéciales
Stabilisé à la lumière, Stabilisé aux intempéries, Stabilisé à la chaleur

Disponibilité régionale
Amérique du nord, Europe, Asia Pacific, South and Central America, Proche-Orient/Afrique

Autres informations

Moulages par injection
Processing conditions:
- Typical melt temperature (Min / Recommended / Max) : 210°C / 220°C / 230°C.
- Typical mold temperature : 10 – 30°C.
- Drying time and temperature (only necessary for bags opened for more than two hours) : 4-8 hours at 60-70°C.

Extrusion (autres)

Processing conditions:
- Typical melt temperature (Min / Recommended / Max): 200°C / 240°C / 270°C.
- Drying time and temperature (only necessary for bags opened for more than two hours): 4-8 hours at 60-70°C.

This database provides the main properties of technical thermoplastics marketed by ARKEMA.

The information contained in this document is based on trials carried out by our Research Centres and data selected from the literature, but shall in no event be held to constitute or imply any warranty, undertaking, express or implied commitment from our part.
Our format specifications define the limit of our commitment.
No liability whatsoever can be accepted by ARKEMA with regard to the handling processing or use of the product or products concerned which must in all cases be employed in accordance with all relevant laws and/or regulations in force in the country or countries concerned.

For information about the other products marketed by our company consult :
ARKEMA

Last update: 2021-02-04 Source: https://www.campusplastics.com
Page: 2/2

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

Annex 3

Biodegradable Polymers

Product Information

Version 1.0
January 2013
G-PMS/B

ecoflex® F Blend C1200

Biodegradable polyester for compostable film

® = ecoflex is a registered trademarks of BASF SE;
Lupolen is a registered trademark of
Lyondell Basell group companies

Product description

ecoflex® F Blend C1200 is our biodegradable, statistical, aliphatic-aromatic copolyester based on the monomers 1.4-butanediol, adipic acid and terephthalic acid in the polymer chain. ecoflex® F Blend C1200 will biodegrade to the basic monomers 1.4-butanediol, adipic acid and terephthalic acid and eventually to carbon dioxide, water and biomass when metabolized in the soil or compost under standard conditions.

ecoflex® F Blend C1200 has properties similar to PE-LD because of its high molecular weight and its long chain branched molecular structure.

BASF
The Chemical Company

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

- Transparent to translucent, semi-crystalline structure with DSC melting point in the range of PE-LD: 110 - 120 °C
- High ultimate elongation at break and high failure energy (dart drop)
- High, but controllable water vapour transmission rate (WVTR)
- MVR (190 °C, 2.16 kg): 2.5 - 4.5 ml/10 min
- Good thermostability up to 230 °C
- regular predrying of pellets
- Good processability on blown film lines
- Down gaging to 10 µm possible
- Weldable and printable

ecoflex® F Blend C1200 fulfils the requirements of the European standard DIN EN 13432, the US standard ASTM D 6400 and the Japanese GreenPla standard for compostable and biodegradable polymers, because it can be degraded by micro-organisms. The biodegradation process in soil depends on the specific environment (climate, soil quality, population of micro-organisms).

ecoflex® F Blend C1200 is one of the few biodegradable plastics, which complies in its composition with the European and American food stuff legislation for food contact: EU Directive 2002/72/EC (as amended) and US food contact notification FCN 907. Specific limitations and more details are given on request. The converter or packer has to check the suitability of the article for the application.

Form supplied and storage

ecoflex® F Blend C1200 is supplied as lens shaped pellets in 1 t big bags or bulk containers. Temperatures during transportation and storage may not exceed 70 °C at any time. Storage time of unopened bags may not surpass 12 month at room temperature (23 °C).

Quality Control

ecoflex® F Blend C1200 is produced as a standard material in a continuous production process according to DIN EN ISO 9001:2000. The melt volume rate, MVR, at 190 °C, 2.16 kg, according to ISO 1133 has been defined as specified parameter for quality control. A certificate can be provided with each lot number (10 t) upon request. In order to obtain a high accuracy for the MVR measurement the granules should be dried for 30 minutes at 70 °C using e. g. an electronic moisture analyser (e. g. Brabender Aquatrac plus). Other data given in our literature are typical values, which are not part of our product specification for ecoflex® F Blend C1200.

Applications

ecoflex® F Blend C1200 has been developed for the conversion to flexible films using a blown film or cast film process. Typical applications are packaging films, agricultural films and compost bags. In view of numerous factors influencing functionality and shelf life of ecoflex® films and finished articles made thereof these parameters have to be tested by the converters before utilisation.

We supply technical service information concerning the blown or cast film process with ecoflex® F Blend C1200 on demand.

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

Intellectual Property

It is the responsibility of those to whom we supply our products to ensure that any proprietary rights and existing laws and legislation are observed. Some uses of ecoflex® and product obtained by use of ecoflex® are subject of intellectual property rights. Purchase of ecoflex® does not entitle the buyer or any third to produce, offer or use any blends of ecoflex® protected under property rights and all their equivalents as listed here:

- EP-B 1656423
- EP-B 937120
- EP-B 950689
- EP-B 1838784
- EP-B 947559
- EP-B 965615

Typical basic material properties of ecoflex® F Blend C1200

Property	Unit	Test Method	ecoflex® F Blend C1200	Lupolen® 2420 F
Mass density	g/cm³	ISO 1183	1.25 - 1.27	0.924
Melt flow rate MFR 190°C, 2.16 kg	g/10 min	ISO 1133	2.7 - 4.9	0.6 - 0.9
Melt volume rate MVR 190°C, 2.16 kg	ml/10 min	ISO 1133	2.5 - 4.5	0.8 - 1.2
Melting point	°C	DSC	110 - 120	111
Shore D hardness	–	ISO 868	32	48
Vicat VST A/50	°C	ISO 306	91	96

Typical properties of ecoflex® F Blend C1200 blown film, 50 µm

Property	Unit	Test Method	ecoflex® F Blend C1200	Lupolen® 2420 F
Transparency	%	ASTM D 1003	82	89
Tensile strength	N/mm²	ISO 527	35/44	26/20
Ultimate strength	N/mm²	ISO 527	36/45	–
Ultimate Elongation	%	ISO 527	560/710	300/600
Failure Energy (Dyna Test)	J/mm	ISO 53373	24	5.5
Permeation rates:				
Oxygen (23°C, dry)	cm³/(m²·d·bar)	ASTM D 3985	1200	2900
Water vapour (23°C, 85% r.h.)	[g/(m²·d)]	ASTM F-1249	135	1.7

Note

The information submitted in this document is based on our current knowledge and experience. In view of the many factors that may affect processing and application, these data do not relieve processors of the responsibility of carrying out their own tests and experiments; neither do they imply any legally binding assurance for a special purpose. It is the responsibility of those to whom we supply our products to ensure that any proprietary rights and existing laws and legislation are observed.

BASF SE
Biodegradable Polymers
67056 Ludwigshafen, Deutschland
www.plasticsportal.eu/ecoflex

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

Annex 4

Lubrizol | ESTANE® ECO 12T80E

Technical Data Sheet

Type: ESTANE® ECO 12T80E is a high-performance bio-based thermoplastic polyurethane.
With Ca 43% bio-based content. Properties similar to standard TPU of same hardness, excellent mechanical properties and abrasion resistance.

Main application: Extrusion/Injection Molding

Physical Properties	Value (Metric)	Unit	Test Method
Hardness	A/1: 82	Shore A/D	ISO 868
Specific Gravity	1.10	g/cm³	ISO 2781
Tensile Strength	33	MPa	ISO 527-2/5A/500
Ultimate Elongation	604	%	ISO 527-2/5A/500
Tensile Stress at:			
- 100 % Elongation	4	MPa	ISO 527-2/5A/500
- 300 % Elongation	9	MPa	ISO 527-2/5A/500
Tear Strength nicked	74	kN/m	ISO 34-1B
Abrasion Resistance	20	mm³	ISO 4649-A
Vicat Softening Point	84	°C	ISO 306 (A50)

* Based on extruded strips

Listed values are "typical (average) values" and should /cannot be applied for specification purposes

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

Lubrizol

ESTANE® ECO 12T80E • Issue date: 07/2021
Issue nr: 1/1

Material Preparation

Prior to processing, **ESTANE® ECO 12T80E** be dried at **100ºC** for 3-4 hours.
It is recommended to dry the material in a dehumidifying type dryer.
Target dew point should be **-40°C.**

Recommended Starting EXTRUSION/MOULDING Temperature Profile:

	°C
Hopper cooling	Yes
Zone 1	190
Zone 2	200
Zone 3	200
Zone 4	200
Adapter 5	200
Die (6)	200
Die Tip (7)	195

For further information refer to Lubrizol Advanced Materials processing guides.

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

Annex 5

Tefabloc™ by mcpp

mcpp Power to Perform

TG SI A06 75A

Product description :
Thermoplastic elastomer partly made of bio–sourced materials
Processing: injection
Product form: granulates (opaque).

Application(s) :
Standard material for:
– Parts subjected to moderate thermal and mechanical stress.
– Soft –touch area for electric boxes, portable phones, sports gear, tools...

Main characteristic(s) :
Good behaviour to U.V.
Broad chemical inertia to
– Base.
– Dish washing powder.
Appearance after processing: rubbery.
Recommended for lively colours.
High flow.
Adapted for bimaterial applications with: PE / PP

Standard compliance :
Material meets requirement:
– European directive 2000 / 53 EC (end of life vehicle)
– European directive 2011 / 65 UE (RoHS: Restriction of the use of certain hazardous substances for electronic and electric devices)

Service temperatures :
Temperature range, without mechanical stress : from –50°C to +100°C
Maximum recommended temperature under stress: +70°C

Processing conditions :
Ready to use
Not hygroscopic and no need for predrying.

RECOMMENDED PROCESSING CONDITIONS
Injection temperatures: Zone 1=200 – 2=210 – 3=220°C
Compression ratio of screw: 2.5 to 3
High injection speed and high screw revolving speed are recommended.
Remark: These parameters provide standard settings and need to be adapted in function of the applied converting equipment.

Fully compatible with PP; hence suitable for recycling with that polymer.
After grinding material can be recycled. Percentage to be adapted in function of material processing restrictions.

Please consult the material safety datasheet before use.

Contacts
EUROPE: France, Western Europe, Africa and other areas: +33 2 51 65 71 43 – Eastern &Central Europe, Scandinavia: +48 46 863 13 60 Asia Pacific: +86 21 6233 4015 – North and South America: +1 888–560–1861

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

Tefabloc by mcpp

mcpp Power to Perform

TG SI A06 75A

Colouring :
Easy coloration with masterbatch for polyolefins.

Packaging – storage :
- 25 Kg PE bags on pallet (film wrapped).
- octabin.
- bigbag.

Store indoors at a temperature below 50°C and avoid direct sunlight.
Despite a longer shelf storage life without loss of properties, we recommend to use the material within 6 months from the delivery date.

Table of properties, please see next page.

Contacts
EUROPE: France, Western Europe, Africa and other areas: +33 2 51 65 71 43 – Eastern &Central Europe, Scandinavia: +48 46 863 13 60 Asia Pacific: +86 21 6233 4015 – North and South America: +1 888–560–1861

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

TG SI A06 75A

Physical mechanical characteristics	Test Method	Units	75A
Hardness, 15 seconds (ShA)	ISO 868	shore A	76
Specific gravity	ISO 1183	g/cm3	0.90
Stress at 20 % elongation	ISO 37	MPa	2.3
Stress at 100 % elongation	ISO 37	MPa	3.1
Tensile strength at break (parallel to the flow)	ISO 37	MPa	3.9
Elongation at break (parallel to the flow)	ISO 37	%	490
MFI 190°C – 2.16 kg	ISO 1133	g/10 min	15.0
Bio–sourced material content	ISO 16620–2:2015	%	40
Values calculated on a limited number of measurements			x

Tested on injected plates.
The figures given are average values and should not be considered as a firm specification.

Please consult the material safety datasheet before use.

Contacts
EUROPE: France, Western Europe, Africa and other areas: +33 2 51 65 71 43 – Eastern &Central Europe, Scandinavia: +48 46 863 13 60 Asia Pacific: +86 21 6233 4015 – North and South America: +1 888–560–1861

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

Annex 6

Tefabloc™ by mcpp

mcpp. Power to Perform

TG SI R02 80A

Product description :
Thermoplastic elastomer partly made of recycled materials, PIR and/or PCR (see data chart) available in a broad hardness range.
SEBS based.
Processing: injection
Product form: Product form: granulates (translucent).

Application(s) :
– Parts subjected to moderate thermal and mechanical stress.
– Soft –touch area for electric boxes, portable phones, sports gear, tools...

Main characteristic(s) :
Broad chemical inertia to
– Acid and base.
– Dish washing powder.
Appearance after processing: rubbery.
Recommended for all colours.
Adapted for bimaterial applications with: PP

Standard compliance :
Material meets requirement:
– European directive 2000 / 53 EC (end of life vehicle)
– European directive 2011 / 65 UE (RoHS: Restriction of the use of certain hazardous substances for electronic and electric devices)

Service temperatures :
Temperature range, without mechanical stress : from –50°C to +100°C
Maximum recommended temperature under stress: +70°C

Processing conditions :
Ready to use
Not hygroscopic and no need for predrying.

RECOMMENDED PROCESSING CONDITIONS
Injection temperatures: Zone 1=200 – 2=210 – 3=220°C
Compression ratio of screw: 2.5 to 3
High injection speed and high screw revolving speed are recommended.
Remark: These parameters provide standard settings and need to be adapted in function of the applied converting equipment.

Fully compatible with PP; hence suitable for recycling with that polymer.
After grinding material can be recycled. Percentage to be adapted in function of material processing restrictions.

Please consult the material safety datasheet before use.

Contacts
EUROPE: France, Western Europe, Africa and other areas: +33 2 51 65 71 43 – Eastern &Central Europe, Scandinavia: +48 46 863 13 60 Asia Pacific: +86 21 6233 4015 – North and South America: +1 888–560–1861

Issue : Ref : 2022-01-17 Text : 2021–02–09

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

Tefabloc by mcpp

mcpp Power to Perform

TG SI R02 80A

Colouring :
Easy coloration with masterbatch for polyolefins.

Packaging – storage :
– 25 Kg PE bags on pallet (film wrapped).
– octabin.
– bigbag.
Store indoors at a temperature below 50°C and avoid direct sunlight.
Despite a longer shelf storage life without loss of properties, we recommend to use the material within 6 months from the delivery date.

Table of properties, please see next page.

Please consult the material safety datasheet before use.

Contacts
EUROPE: France, Western Europe, Africa and other areas: +33 2 51 65 71 43 – Eastern &Central Europe, Scandinavia: +48 46 863 13 60 Asia Pacific: +86 21 6233 4015 – North and South America: +1 888-560-1861

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

TG SI R02 80A

Physical mechanical characteristics	Test Method	Units	80A
Hardness, 15 seconds (ShA)	ISO 868	shore A	81
Specific gravity	ISO 1183	g/cm3	0.89
Tensile strength at break (parallel to the flow)	ISO 37	MPa	9.4
Elongation at break (parallel to the flow)	ISO 37	%	670
MFI 230°C – 2.16 kg	ISO 1133	g/10 min	23
Recycled material content (PCR et/ou PIR)	ISO 14021	%	66
Values calculated on a limited number of measurements			x

Tested on injected plates.
The figures given are average values and should not be considered as a firm specification.

Please consult the material safety datasheet before use.

Contacts
EUROPE: France, Western Europe, Africa and other areas: +33 2 51 65 71 43 – Eastern &Central Europe, Scandinavia: +48 46 863 13 60 Asia Pacific: +86 21 6233 4015 – North and South America: +1 888–560–1861

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

Annex 7

The LYCRA Company

PRODUCT INFORMATION FOR GENERAL RELEASE MARCH 4, 2019

LYCRA® T275B and T275L fibers

LYCRA® lastingFIT technology for swimwear

Stubborn Chlorine Resistance

With a premium level of chlorine resistance, LYCRA® T275B and T275L fibers are the innovations behind LYCRA® lastingFIT technology.

Swimwear fabrics with LYCRA® lastingFIT technology offer long-lasting fit in chlorinated water environments: up to 10 times longer than fabrics made with unprotected elastane. They also provide the comfort and free-movement for which the LYCRA® brand is famous worldwide.

Features of LYCRA® T275B and T275L Fibers

- **Chlorine resistance.** Prolonged power retention to help swimwear keep its shape in the pool.
- **Uniformity.** Excellent fabric uniformity, particularly in combination with synthetic filament yarns.
- **Contains no heavy metals.** Chlorine resistance without heavy-metal additives.
- **Comfort.** The comfort that consumers expect from quality swimwear with LYCRA® fiber.
- **Bright luster (T275B) or delustered (T275L).** Helps to prevent elastane glitter and yellowing.

Power Retention in Chlorinated Water

In chlorinated water all elastane fibers tend to lose retractive power over time, resulting in a loss of garment shape or "bagging". The amount of time an elastane endures before a significant loss of power occurs (i.e., about 40% power loss) determines the usable life of a garment in terms of shape retention.

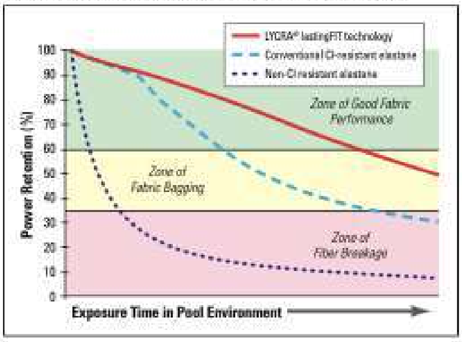
Pool tests performed by The LYCRA Company measure the power loss over time of swimwear fabrics that are continually exercised in a simulated swimming pool environment. Figure 1 illustrates test results for swimwear fabrics with different elastane yarns but otherwise having identical construction. The figure shows that swimwear fabrics with LYCRA® lastingFIT technology have a demonstrated ability to retain more than 60% of their original power for an extended period:

- for about twice as long as fabrics with conventional chlorine-resistant elastane.
- for about 10 times as long as fabrics containing non-chlorine resistant elastane.

Typical Properties

Yarn Size (dtex)	22, 33, 44, 60, 78, 117, 156
Luster	Bright or delustered
Elongation at Break	~490% (44 dtex)
Load Power at 200% Elongation	~6.4 cN (44 dtex)
Tenacity (cN per dtex)	~0.9 (44 dtex)

Figure 1. Chlorine resistance of swimwear fabrics.



This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

Packaging

LYCRA® T275B and T275L fibers are provided on individual tubes or pre-warped beams.

Dtex	Size	Put-up	Source	Luster
22	450 g	NT	Singapore	B
33	600 g	NT	South America	L
33	450 g	NT	Singapore	B
44	600 g	NT	North America	B
44	600 g	NT	South America	L
44	600 g	NT	Singapore	B
60	650 g	NT	North America	B
60	600 g	NT	Singapore	B
78	600 g	NT	Singapore	B
78	600 g	NT	South America	L
117	600 g	WT	Singapore	B
156	1100 g	WT	Singapore	B
156	1200 g	WT	North America	B

Key to lusters: B = bright, L = delustered

NT = Narrow Tube (58 mm length)
WT = Wide Tube (116 mm length)
All tubes are 74 mm interior diameter.

Processing Information

Dyebath pH can affect the longevity of chlorine resistance in any elastane fiber, including LYCRA® T275B/L fiber. Exposing this fiber to a pH below 4.5 can significantly deplete chlorine resistance.

LYCRA® T275B/L fiber can be heat-set at temperatures ranging from 188 to 199°C (370 to 390°F) at exposure times of up to 60 seconds. Experiment for best results.

LYCRA® T275B/L fiber is compatible in processing when blended with cellulosics, polyester, or polyamide. It is not degraded by any dye class now in commercial use. LYCRA® T275B/L fiber has the same dye affinity as other LYCRA® elastane fibers, and should require no major changes in dye formulations having acid, direct, fiber reactive, and disperse dyes versus other LYCRA® elastane fibers.

The use of chlorine-liberating chemicals, unsaturated fatty acids, unsaturated fatty esters, and unsaturated fatty amines should be avoided during wet finishing as these tend to discolor and degrade elastane fibers.

Branding

Fabrics that are elastified with LYCRA® T275B/L fiber, and which meet the LYCRA® fiber quality standards for swimwear, qualify for the **LYCRA® XTRA LIFE™** brand. Contact your representative at The LYCRA Company for more information.

LYCRA xtra life™

Visit us on the web at: connect.lycra.com

The LYCRA Company innovates and produces fiber and technology solutions for the apparel and hygiene industries, as well as specialty chemicals used in the spandex and polyurethane value chains. Headquartered in Wilmington, Delaware, The LYCRA Company is recognized worldwide for its innovative products, technical expertise, and unmatched marketing support. The LYCRA Company owns leading consumer and trade brands: LYCRA®, LYCRA HyFit®, LYCRA® T400®, L by LYCRA®, COOLMAX®, THERMOLITE®, ELASPAN®, SUPPLEX®, TACTEL®, and TERATHANE®. While The LYCRA Company's name is new, its legacy stretches back to 1958 with the invention of the original spandex yarn, LYCRA® fiber. Today, The LYCRA Company is focused on adding value to its customers' products by developing unique innovations designed to meet the consumer's need for comfort and lasting performance. For more information, visit connect.lycra.com and lycra.com.

Doc Ref: P030

The LYCRA Company

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.

Annex 8

Group4 : Haute élongation (Ténacité modifiée)

	Clamp type	With twister and bollards; 4k
	Load cell	100 N
	Automatic threading	☑
- Automatic threading options		
	Package change inside group	☑
	Package change after group	☑
	Changer back to position 1 after last test of all groups	☑
- Yarn handling during threading		
	Nip roller closed and passive	◉
	Nip roller closed and active	☐
	Nip roller open	☐ Only without use of twisting device!
	Slow threading arm	☐
	Insert yarn in twister	1 times
- Yarn handling during testing		
	Air flow during test	☐
- Yarn handling during yarn clearing		
	Clear with nip roller	100 %
	Number of cuts	3 n
+ Nip roller transport at start of package		
+ Count test with AUTOCOUNT		
- 4. Standard tensile test		
	Gauge length	180 mm
	Test speed	250 mm/min
	Pretension	.5 cN/tex
- Bollards		
	Use bollards	☐
- Twisting device		
	Use twisting device	☐
- Identification of thread breakage		
	Threshold	.1 % of 100 N
	Drop of force	40 %
- Yarn clearing/loading		
	Blow off	☑
	Break all filaments	☑
- Nip Roller device		
	Transport after n tests	☐

This project has received funding from the Horizon 2020 Programme under grant agreement n°101003906.