



AQUAFIL 
synthetic fibres and polymers

- Aquafil Group Worldwide (plants location, manufacturing processes, products, market application)
- BCF B.U.- R&D Activity
 - Organisation & Skill
 - Research Project approach
 - Collaboration with UNITN and other reseach centre
 - R&D running projects
 - Process and technology transfer activity

Aquafil at a glance



€ 507 million – turnover 2014

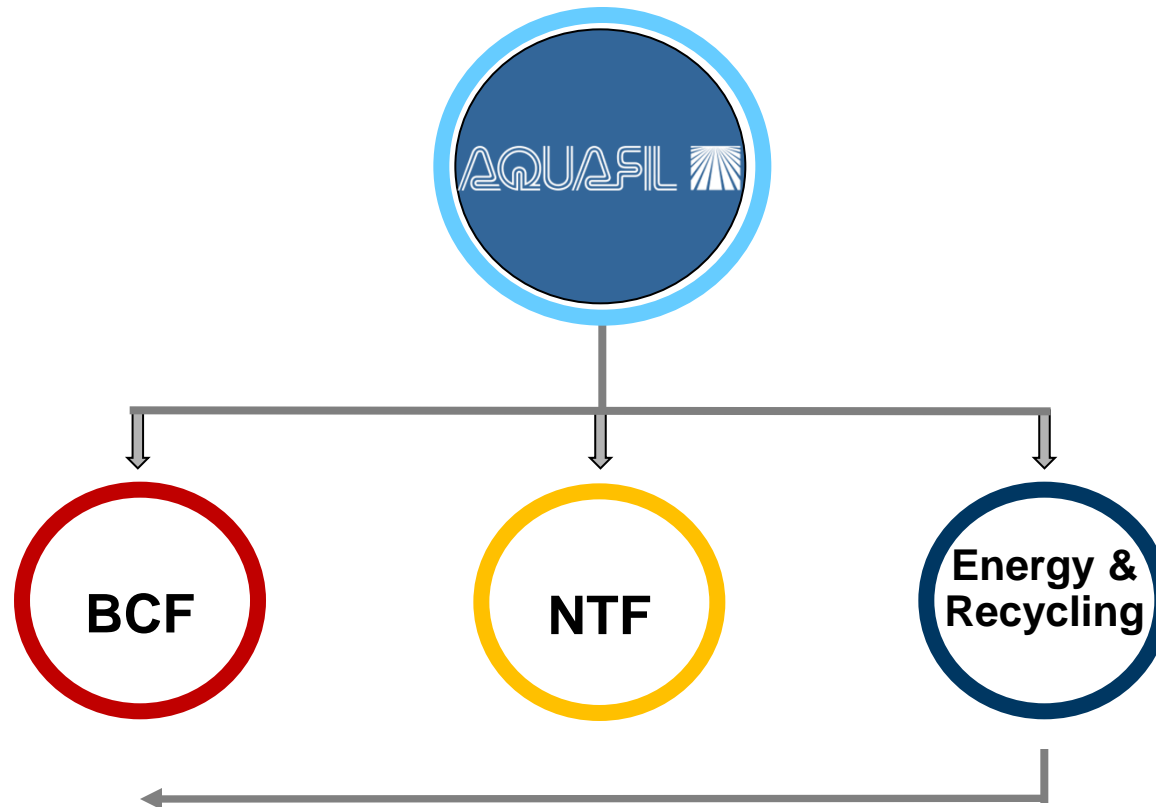
15 Plants (4 in Italy)

2791 employees

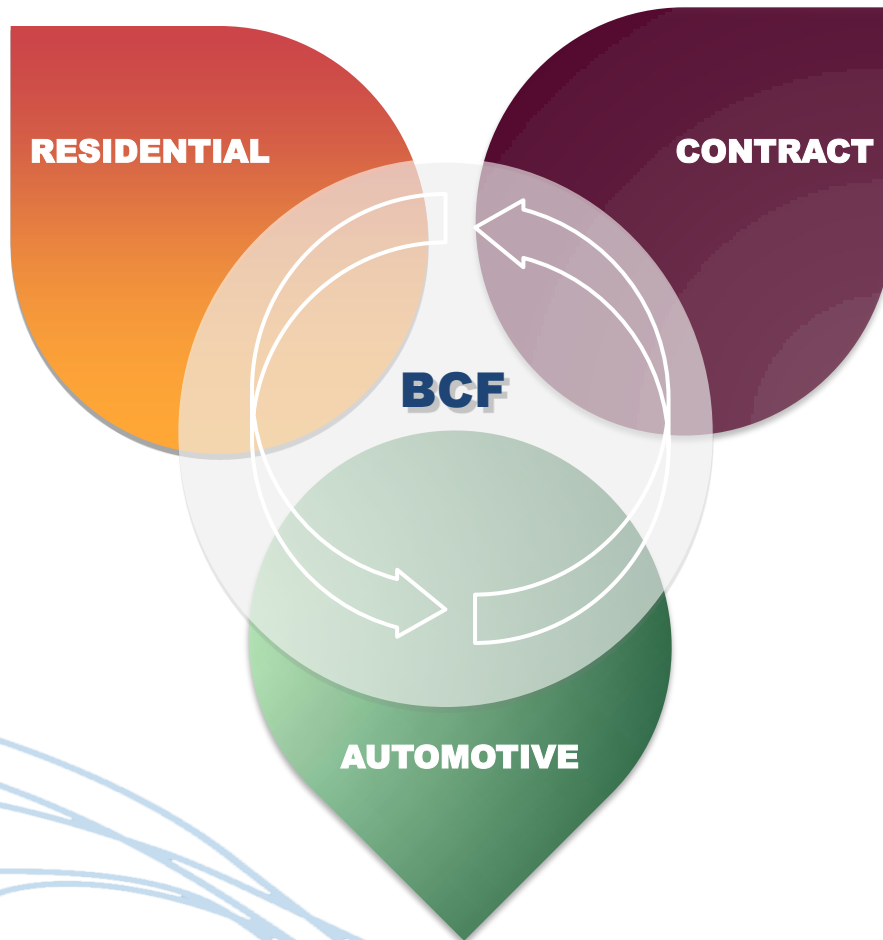
130,000 tons of polymers and fibers produced every year

- YARN FOR CARPET
- YARN FOR GARMENT
- WASTE WATER TREATMENT - Aquaspace, Rovereto
- ENGINEERING, Aquafil Engineering, Berlin

Our business units



BCF business unit (Bulk Continuous Filament)



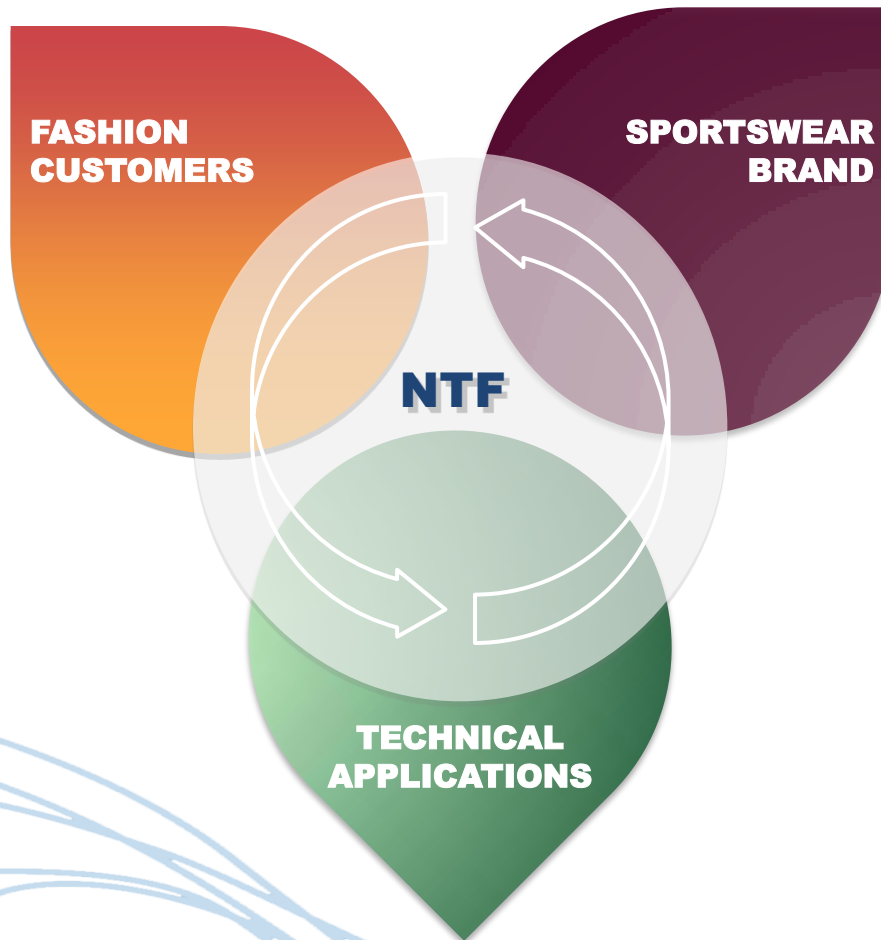
ECONYL®
Made with 100% Regenerated Nylon

 **ALTO**
AQUAFIL

 **ALTO**
CHROMA
AQUAFIL

 **AQUALON**
AQUAFIL

NTF business unit (Nylon Textile Filament)



ECONYL®
Made with 100% Regenerated Nylon

ultralon®
BY AQUAFIL

MicroLON®
BY AQUAFIL

Borgolon®
BY AQUAFIL

THE FEEL GOOD MICROFIBRE
Dryarn®
BY AQUAFIL



BU Bcf



BU Ntf

BU Energy & Recycling

THE **ECO**
PLEDGE®



Aquafil's Path toward Full Sustainability



Energy



Recycling



Culture of
sustainability





- From cogeneration to trigeneration
- Green certified energy
- Solar power

ENERGY – The cogeneration plant



Opened in 2006 in Arco plant, Italy. In 2008 we improved it from cogeneration to trigeneration.

The system has a nominal electrical output of 8.8 Mwe and a thermal capacity of 9Mwt.

From 2006 to 2013
the plant has achieved
a total saving of 27,103 TEP



ENERGY – Certified green energy



Since the start in 2010, the Aquafil Group's **Italian sites** have only purchased energy produced from renewable sources which prevents the emission of more than 55,000 tons of CO₂.

The origin of that energy is guaranteed by the European RECS (Renewable Energy Certificate System) label.

From 2013 to 2015 in **Croatia** – 100% energy from renewable sources

From 2013 in **Slovenia** – 22% energy from renewable sources (hydroelectric, eolic and others)



ENERGY – Solar Power Energy



- Arco solar power plant, Italy
CO2 avoided 286 ton/year
- Tessil4 roof, Cares, Italy
CO2 avoided 350-400 ton/year
- Cartersville solar power plant, USA
CO2 avoided 364 ton/year





- Recycling
- The ECONYL® Plant

RECYCLING – A long journey



Recovery of the
lactamic water

1990



Recovery of
internal PA6
discard

1998



Pre-consumer
discard

2007



Post-consumer
discard

2011

RECYCLING – The Econyl® Plant

Four years and over 20 million euro have been spent on the R&D, design and commissioning of the chemical system and depolymerization plant (not counting the already existing infrastructures)



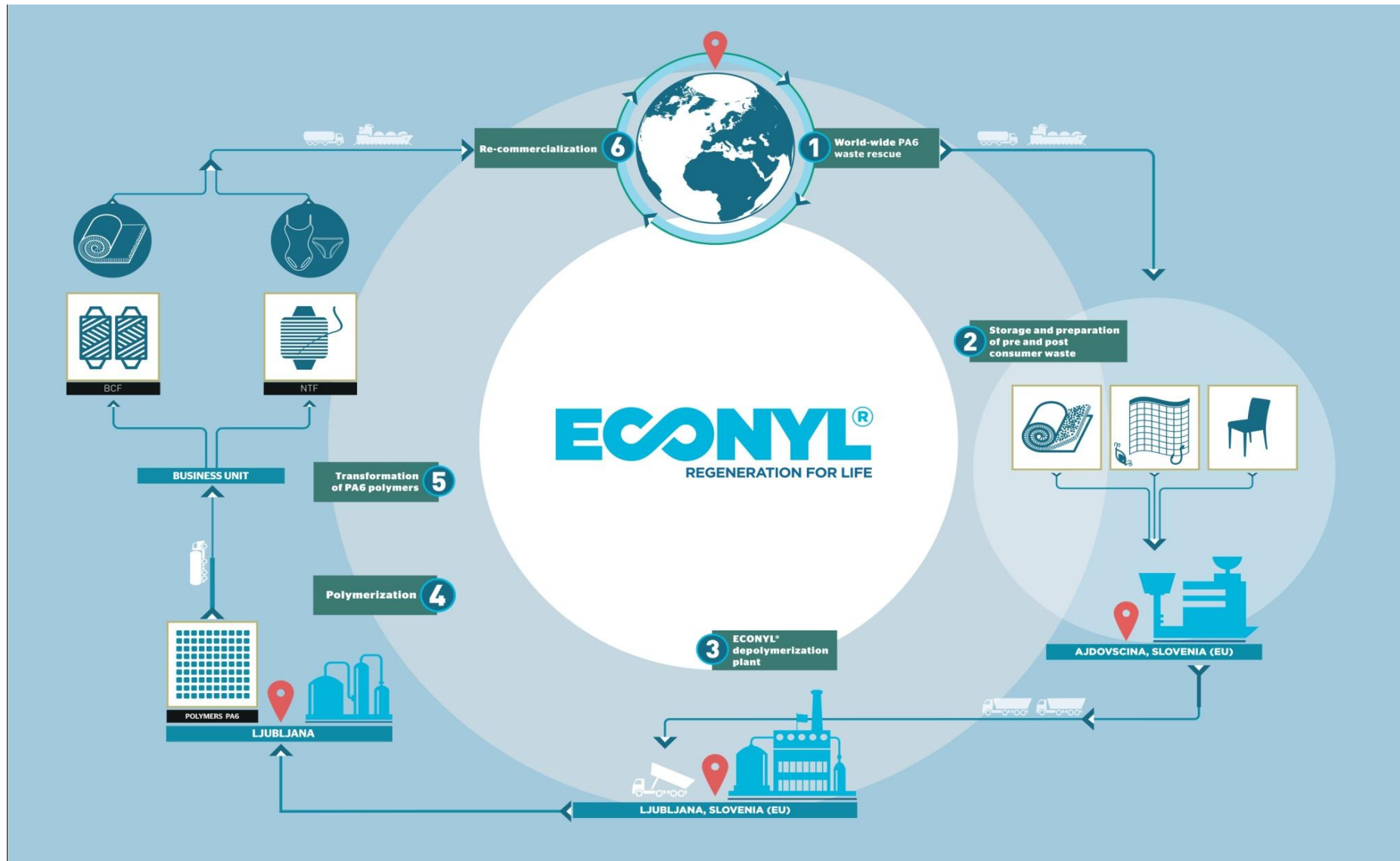
RECYCLING

Target for 2015: 25,000 tons of ECONYL® yarn

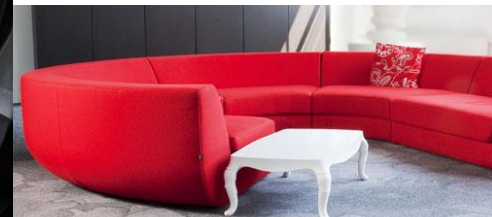
We substitute fossil raw materials

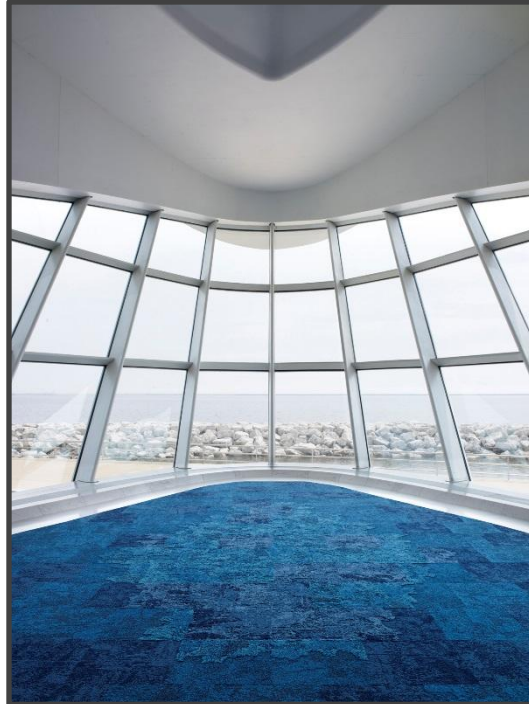
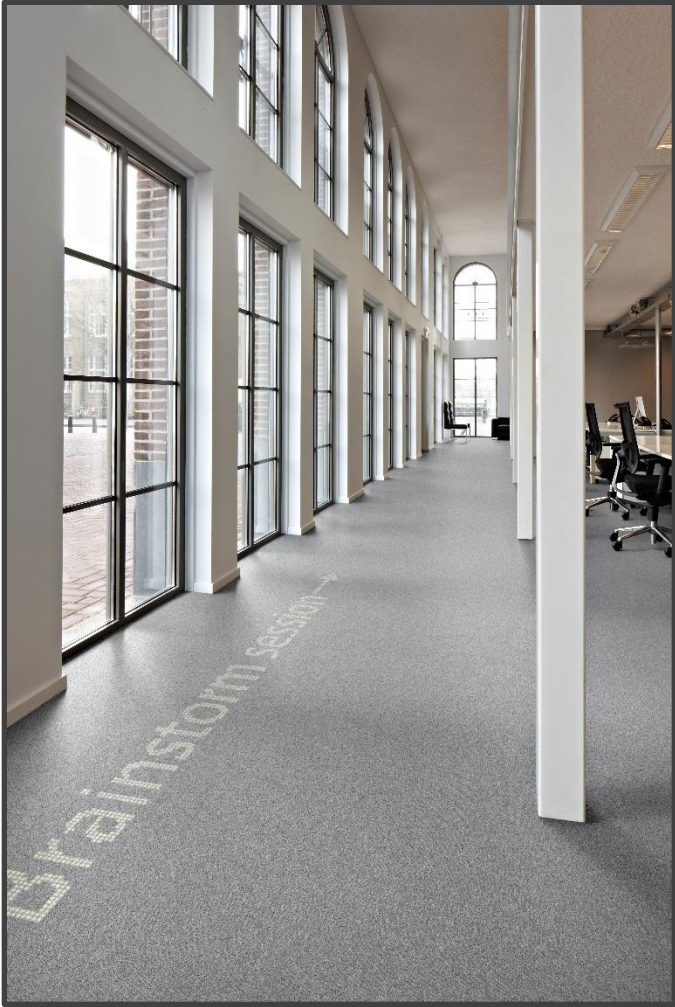
Crash Course 06_04_2015

THE ECONYL® REGENERATION SYSTEM



Products





ECONYL®
REGENERATION FOR LIFE

adidas®

arena®

LA PERLA

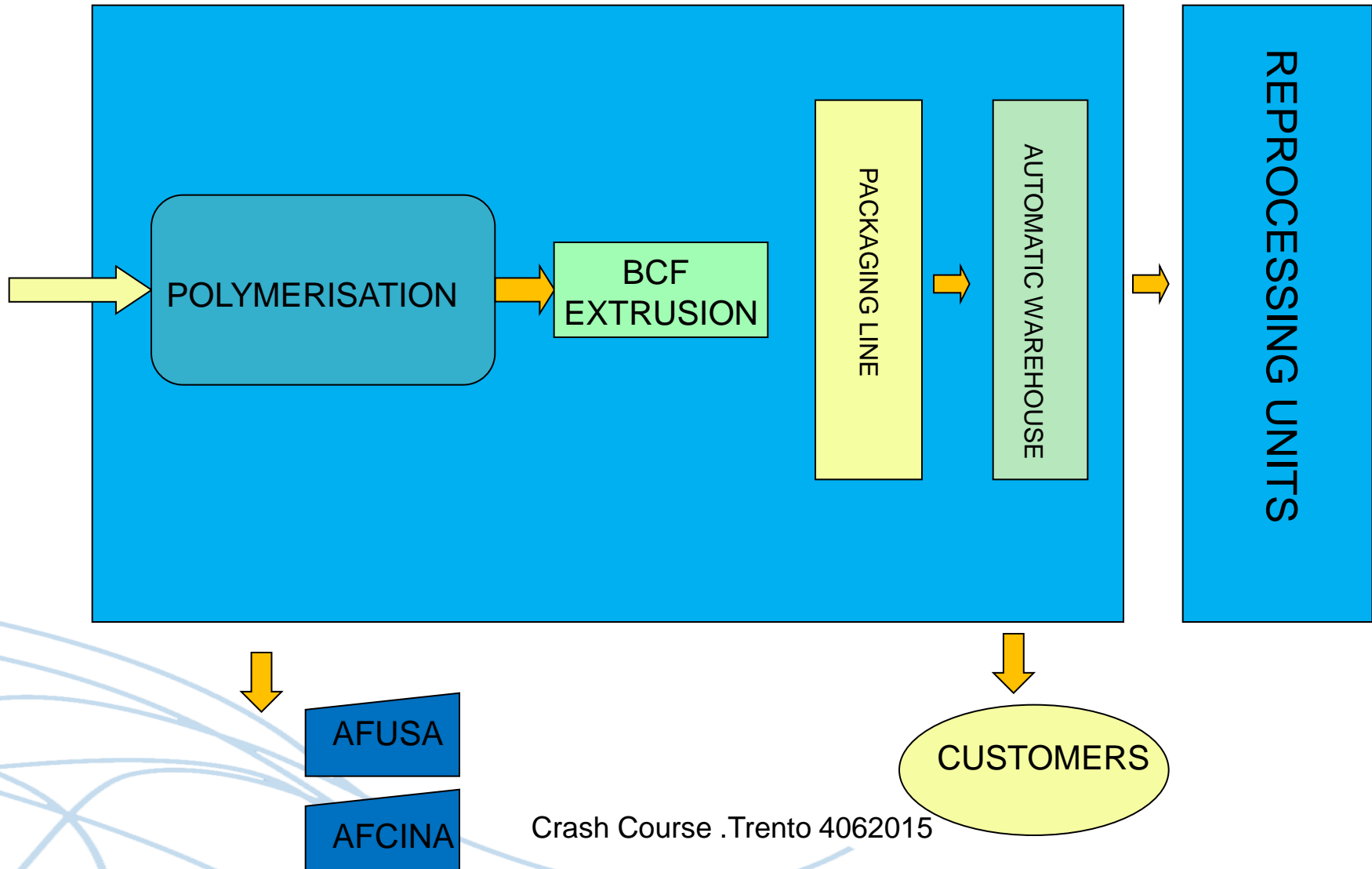
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tshirt project for Econyl(R)

Wave-o
by Ondine de La Feld

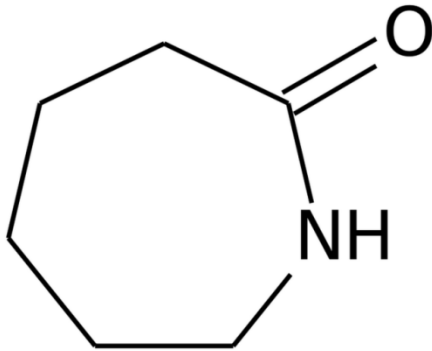
BCF production chain



Polymerisation process

From caprolactam to nylon 6

- Nylon 6 was developed from Paul Schlack in 1937 by IG Farben, from hydrolitic polymerisation of caprolactam



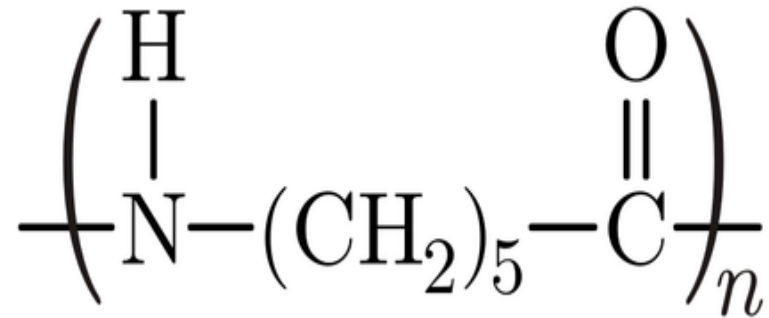
Name: caprolactam (CPL)

Molecular formula: C₆H₁₁NO

Molecular mass: 113,16

Density [g/cm³]: 1,01

Melting point [° C]: 69



Name: Nylon 6 (PA6)

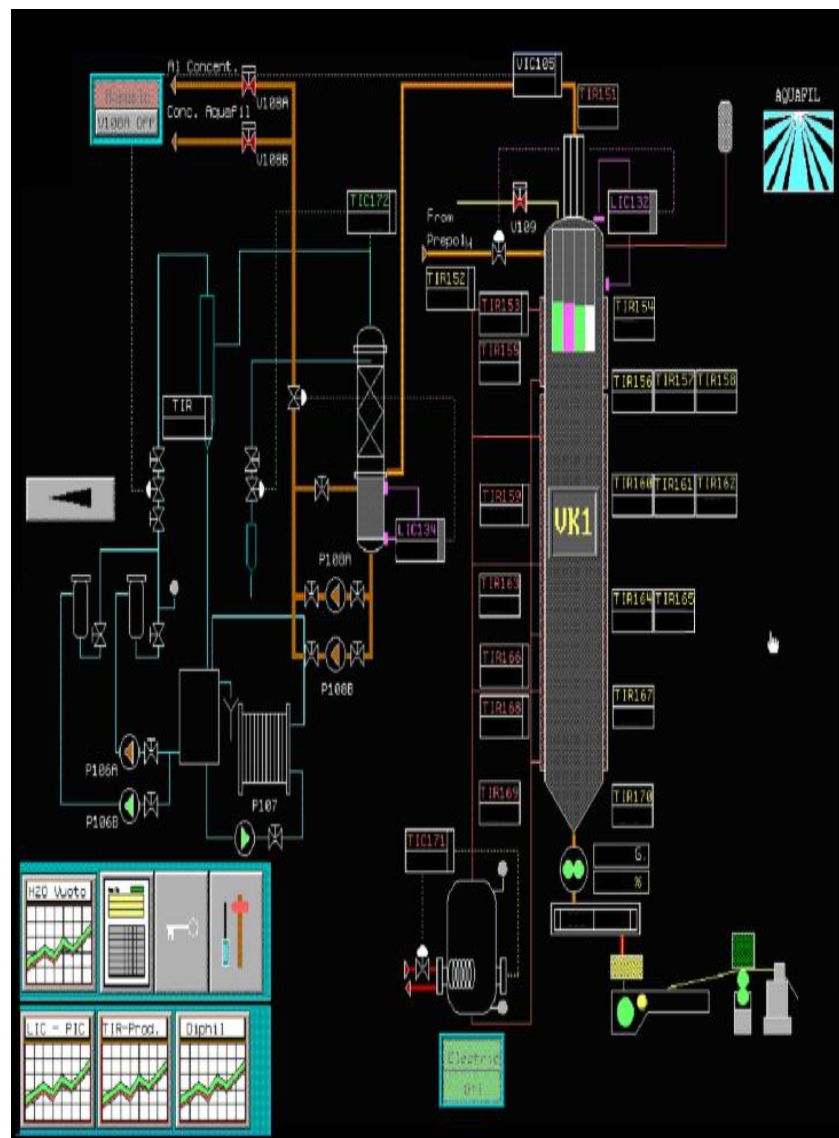
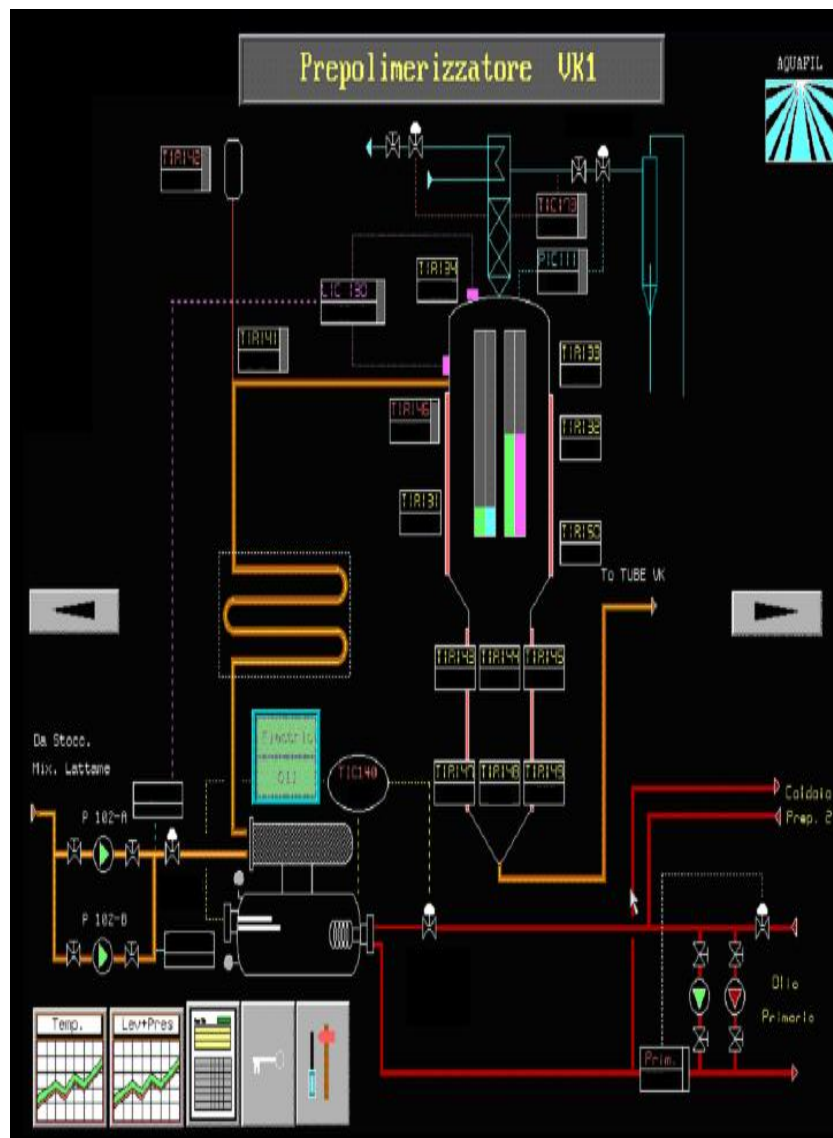
Molecular formula: (C₆H₁₁NO)_n

Molecular mass: 15.000-25.000

Density [g/cm³]: 1,14

Melting point [° C]: 220-230

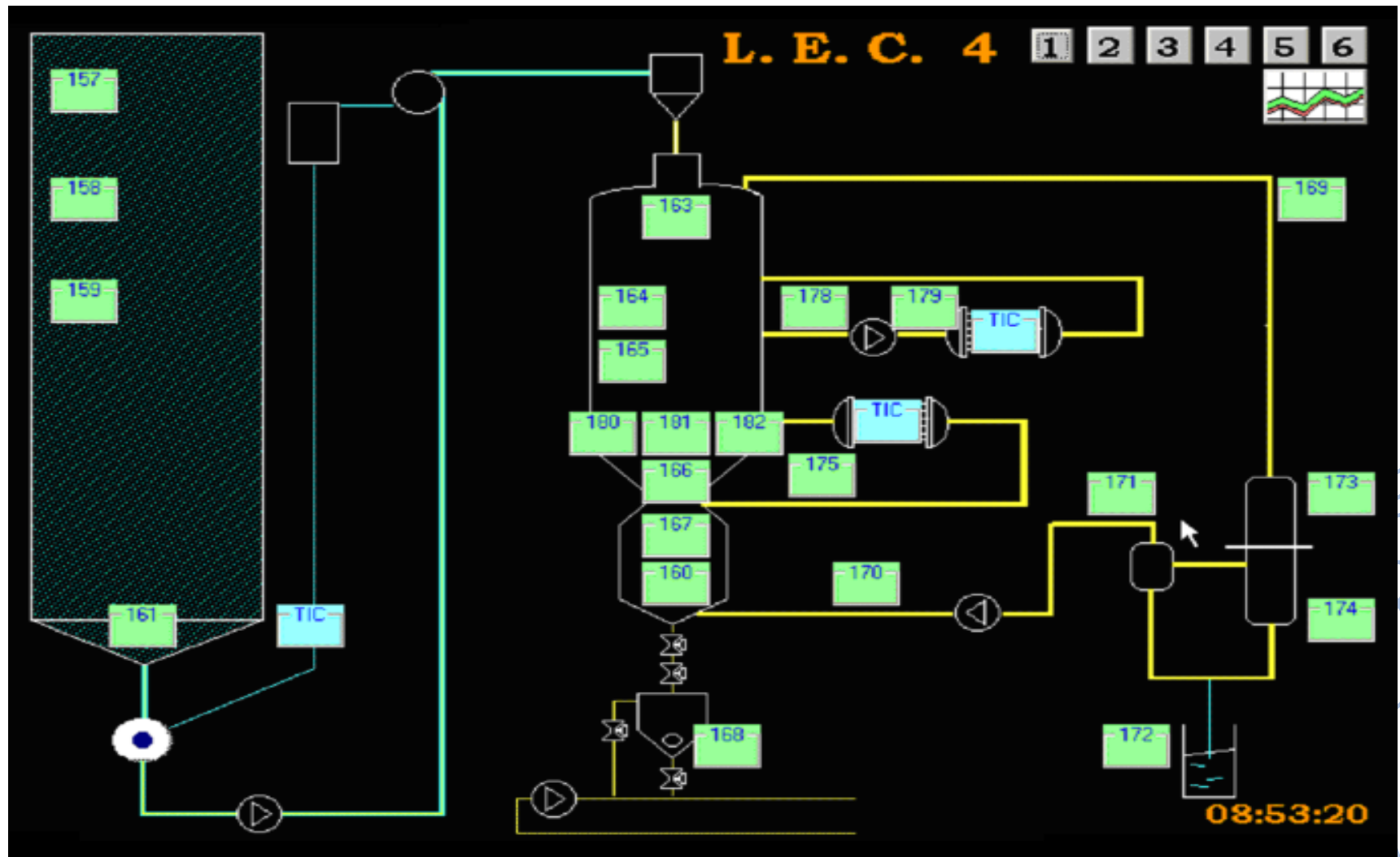
Polymerisation – continuous process



Polymerisation – continuous process



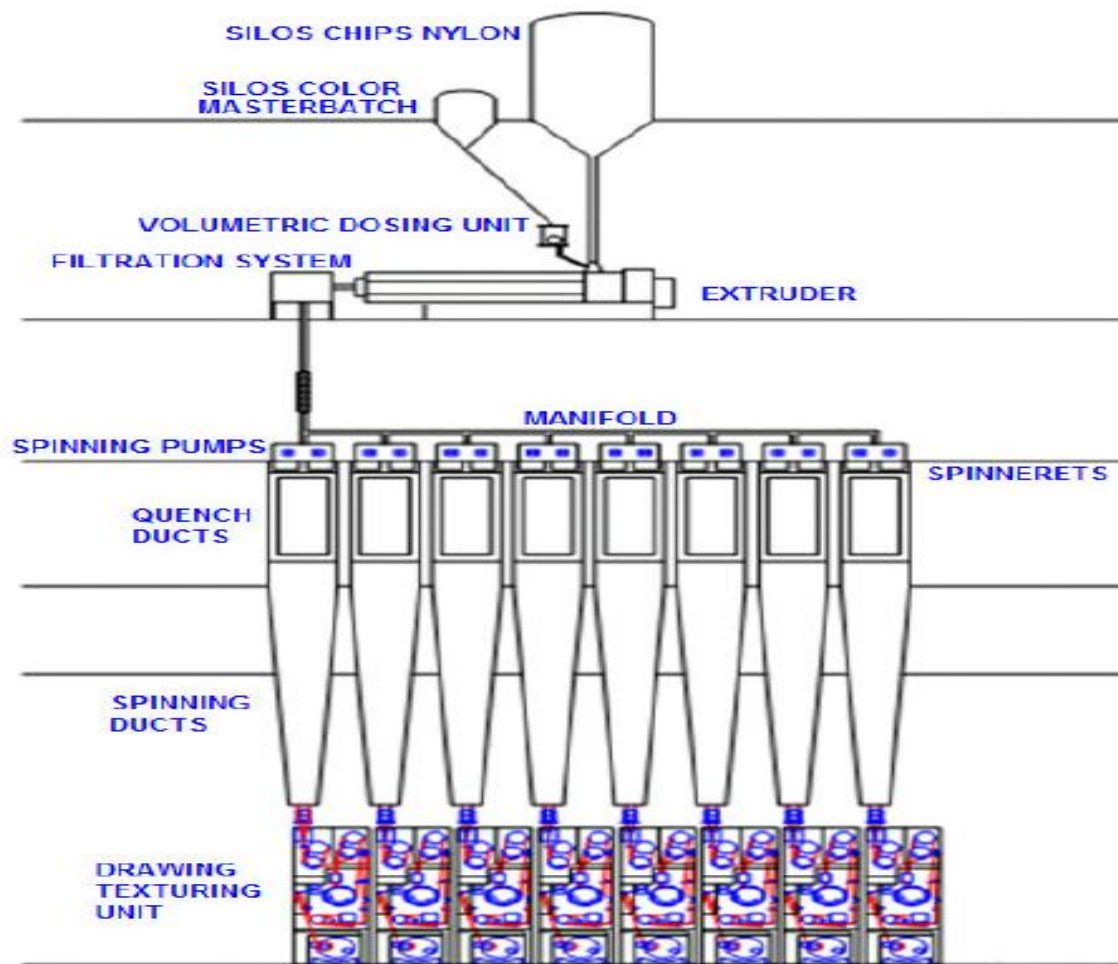
Continuous washing & drying

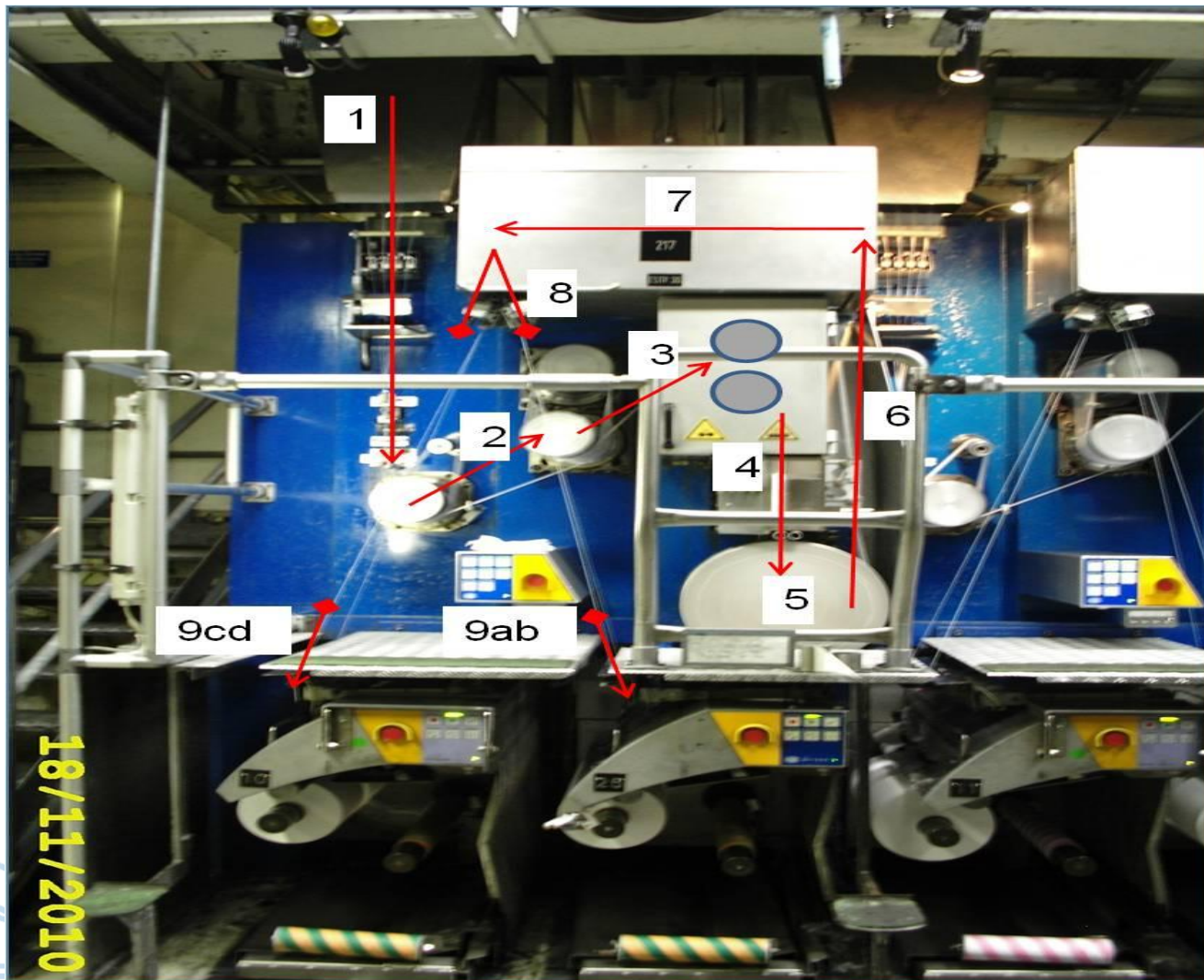


From polymerisation to extrusion



Scheme of BCF spinning line





R&D activity

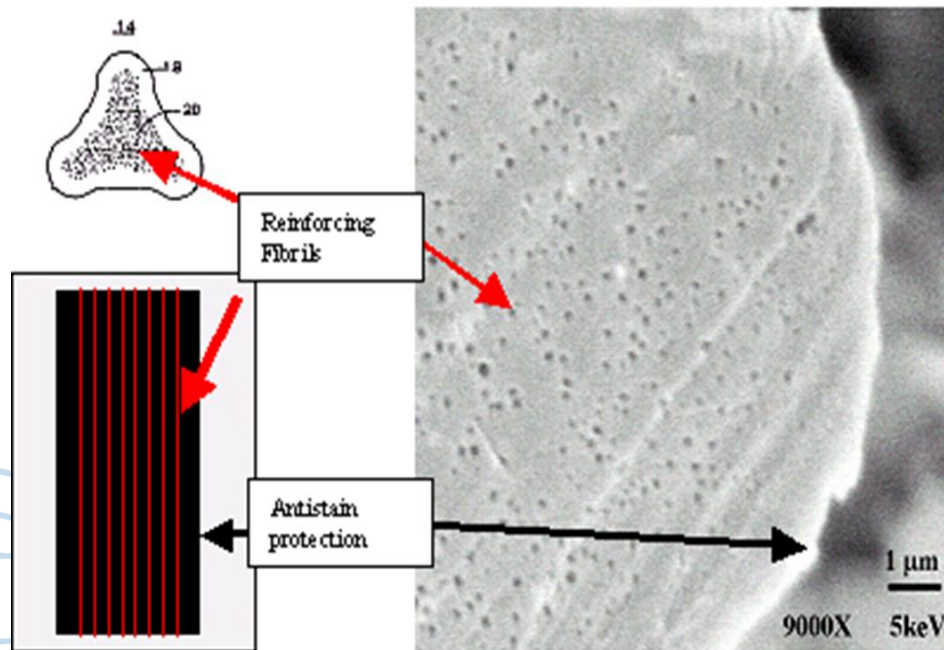
Structure and organization

R&D activity, focused and oriented to research projects, process improvement, product upgrade is carried out in Arco location since:

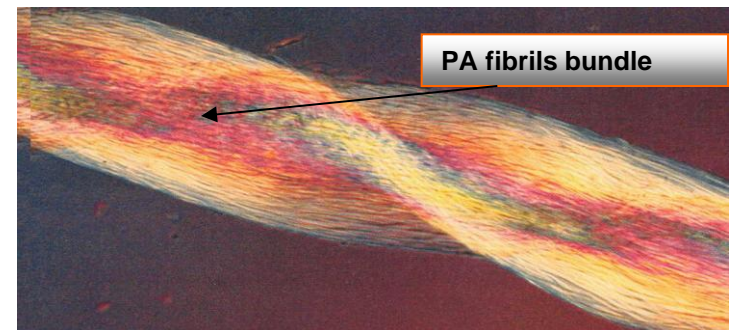
- There is a full availability and support of an off-line pilot plants (autoclave, spinning line, depolymerisation and CPL distillation line)
- Very well equipped CQ and Research chemical lab (DSC, FT-IR, two GC, UV spectrophotometer, Rheometer, HPLC) and textile lab (Dynamometer, NMR for spin finish determination, Xenotest (2), colour lab Spectrophotometer (3) for colour measure and colour matching)
- Very good, professional and skilled team of material & chemical engineers fully committed and research and process oriented

Research projects: what is done

- Aquafil has a very long experience in managing research projects using funds coming from different sources (Local authorities , European funds , self financed)
- Project MTX “Development of an innovative fibre for floorcovering application” - 2001-2005



- Reactive extrusion
- Compatibilization between PP and PA
- Creation of a” composite- like
- “structure



- Project AUTOCar “High Performance Fibres for Automotive ”
2006/2009

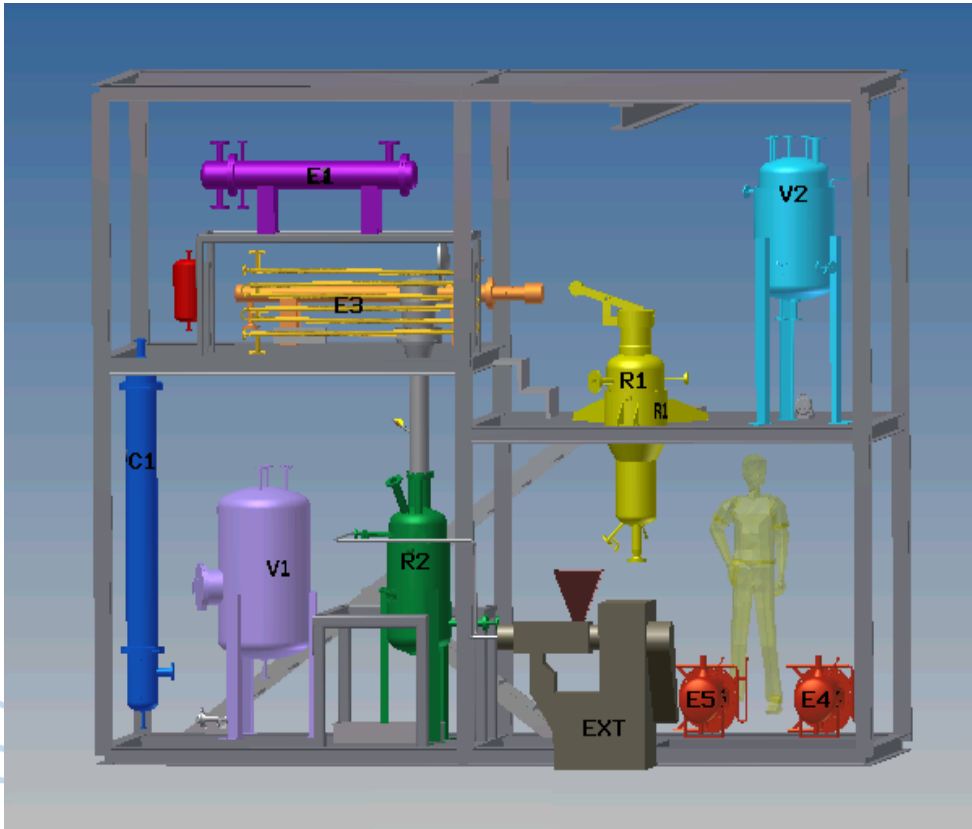


Development of a high performing yarn as a full package:

- Development of a low staining polymer
- High performing yarn with high bulk and low weight
- Enhanced yarn crystallinity

Research projects: what is done

➤ Project NEXTOR “Never Ending Story of PA6”-2009/2014



- Design and construction of pilot plant for recycle
- Claiming, selection , identification of PI & PC goods PA6 based
- PI PC cleaning , grinding and compacting
- Recycled to PA6 through physical (re-extrusion and compounding process)
- Regeneration to the original CPL through depolymerisation and distillation
- LCA evaluation of the whole process

Research projects: what is done

- Project BRACE : “ Better resistance across clay ‘s evolutions ”
AquaFil-UFI-Filter -2010/2012



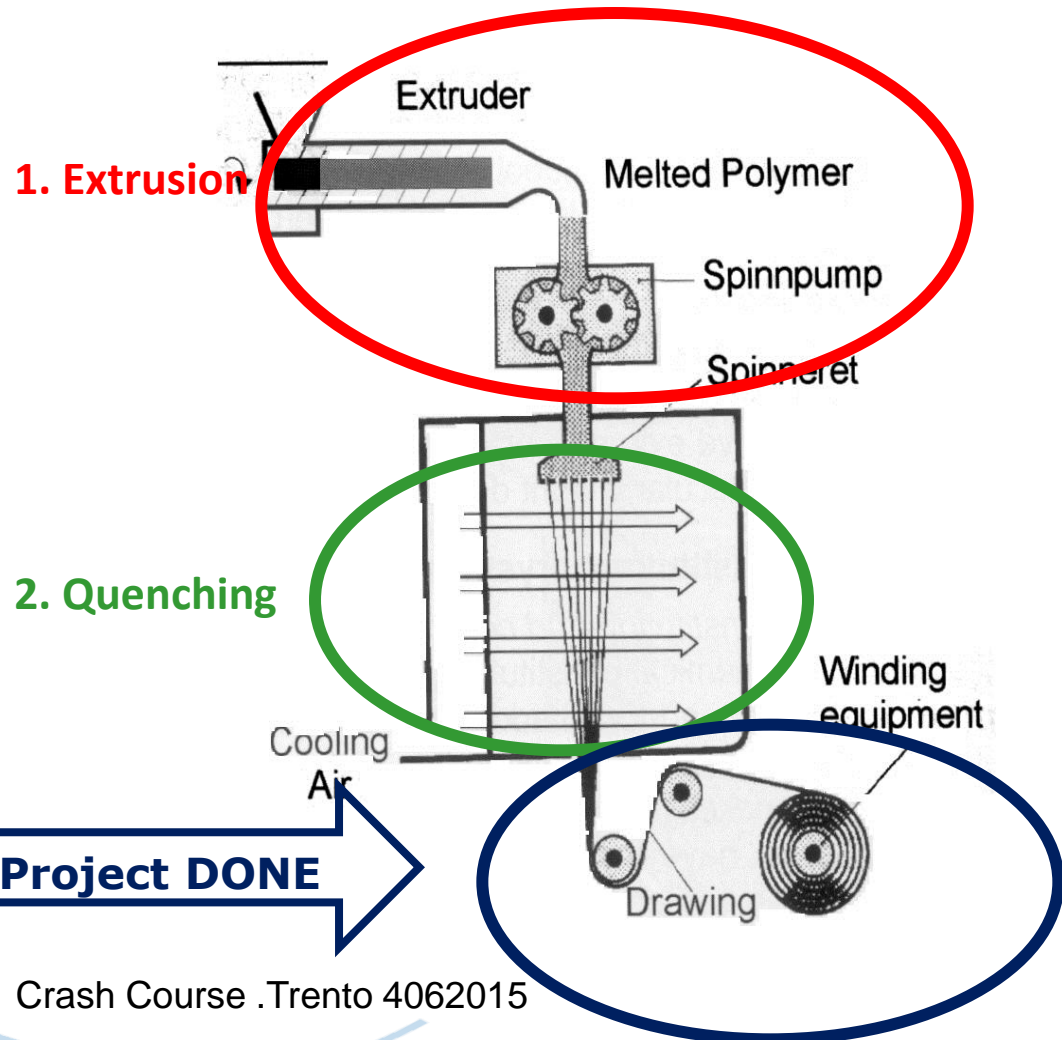
Support in PA66 GF

Filtering Cartridge in non woven PA6

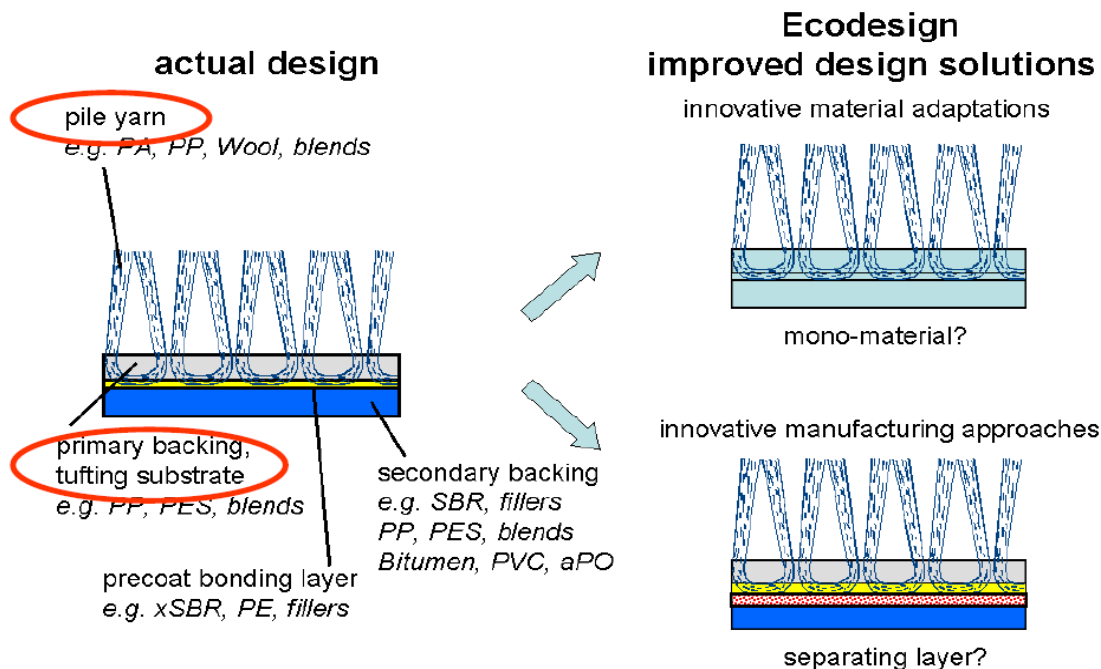
Development of a PA6 with nanoparticles (bentonite) to produce a single component filtering cartridge, replacing the combination PA66 support + standard PA6 filter

Research projects: what is going on

- Project DONE "Development Optimal New Entanglement"- 2013 still ongoing



- Project EcoMeTex-Ecodesign Methodology for recyclable Textile covering- Seven Framework Programme – 2012 ongoing



Design and construction of an easily recyclable carpet with advantages for the environment.

In collaboration with all the carpets productive chain and with some research centres.

- PCT WO 03/029536 A1 Improved polyolefine –based synthetic fibers and methods therefor 10 April 2003 or EP 1446518 B1 28 January 2001
- Bisfa Generic Name

DIRECTIVES

COMMISSION DIRECTIVE 2011/73/EU of 29 July 2011

amending, for the purposes of their adaptation to technical progress, Annexes I and V to Directive 2008/121/EC of the European Parliament and of the Council on textile names

(Text with EEA relevance)

THE EUROPEAN COMMISSION,

(1) in Annex I the following row 49 is added:

Having regard to the Treaty on the Functioning of the European Union,

Having regard to Directive 2008/121/EC of the European Parliament and of the Council of 14 January 2009 on textile names ⁽¹⁾, and in particular Article 15(1) thereof,

Whereas:

- (1) Directive 2008/121/EC lays down rules governing the labelling or marking of products as regards their textile fibre content, in order to ensure that consumer interests are thereby protected. Textile products may be placed on the market within the Union only if they comply with the provisions of that Directive.

'49.	Polypropylene/polyamide bicomponent	a bicomponent fibre composed of between 10 % and 25 % by mass of polyamide fibrils embedded in polypropylene matrix';
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(2) in Annex V the following entry 49 is added:

'49.	Polypropylene/polyamide bicomponent	1,00'.
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- **Processabilità di miscele PA6 con poliestere per filature**-Tesista Denis Lorenzi; Relatore Prof. Luca Fambri-Tesi di Laurea in Ingegneria dei materiali presso la Facoltà di Ingegneria dell'Università di Trento;Anno Accademico 2006 – 2007.
- **Filatura e stirabilità di omopolimeri e di miscele**-L. Fambri, D. Lorenzi, L. Comperatore, L. Bertamini, G. Giacomelli-AIM XVIII Convegno Nazionale di Scienza e Tecnologia delle Macromolecole(Catania, 16 – 20 settembre 2007)
- **Spinnability and Drawability of Polypropylene, Polyamide 6 and Their Blends**-D. Lorenzi, A. Casagrande, E. Pasqualini, G. Giacomelli, M. Caldara, L. Fambri-Trento Innovation Conferences in Materials Engineering – Advances in Polymers, Composites and Biomaterials(Trento 16 -19 December 2007)
- **Miscela PBT/PA6 per filature**-D. Lorenzi, C. Gavazza, M. Caldara, G. Giacomelli, E. Pasqualini, L. Fambri-AIM XIX Convegno Nazionale di Scienza e Tecnologia delle Macromolecole-(Milano, 13 – 17 settembre 2009)

Conference contributions:

- **24th International Symposium on Polymer Analysis and Characterization** June 6-8th, 2011, Torino (Italy) ***“Rheological and Molecular Characterization of regraded Nylon-6 obtained in Different Reactive Compounding”*** L.Fambri, M.Buccella, A.Dorigato, E.Pasqualini, M.Caldara
- **XX Convegno Italiano di Scienza e Tecnologia delle Macromolecole** September 4-8th 2011, Terni (Italy) ***“Rigradazione di poliammidi industriali: caratterizzazione reologica e termo-meccanica”*** M.Buccella, A.Dorigato, E.Pasqualini, G.Giacomelli, M.Caldara, L.Fambri
- **European Polymer Federation Congress 2013** 16-21st June 2013, Pisa (Italy) ***“Regradation of industrial polyamide 6. Comparison of different chain-extendors.”*** M.Buccella, A.Dorigato, M.Caldara, L.Fambri
- **Preparation of nanofilled polyamides for spinning and drawing**, Dresden 21-25 August 2011, Fambri, Lorenzi, Buccella, Giacomelli, Comperatore
- **Third International Conference of Multifunctional , Hybrid and Nanomaterials**, 3-7 March 2013, Sorrento ***“Effect of different nanofiller in polyamide 6 “***, Fambri, Pegoretti, Lorenzi, Dorigatti, Giacomelli, Brugnara

International Scientific Journal:

- M.Buccella, A.Dorigato, E.Pasqualini, M.Caldara, L.Fambri “***Thermo-mechanical properties of Polyamide 6 chemically modified by chain extension with Polyamide 6-Polycarbonate blends***”, Journal of Polymer Research, 2012, 19(8), 9935-9943. DOI: 10.1007/s10965-012-9935-02012.
- M.Buccella, A.Dorigato, M.Caldara, E.Pasqualini, L.Fambri “***Thermo-mechanical behaviour of Polyamide 6 chain extended with 1,1'-Carbonyl-Bis-Caprolactam and 1,3-Phenylene-Bis-2-Oxazoline***”, Journal of Polymer Research, 2013, 20(9), 1-9. DOI: 10.1007/s10965-013-0225-2
- M.Buccella, A.Dorigato, E.Pasqualini, M.Caldara, L.Fambri “***Chain extension behaviour and thermo-mechanical properties of Polyamide 6 chemically modified with 1,1'-Carbonyl-Bis-Caprolactam***”, Polymer Engineering and Science, 2013. DOI: 10.1002/pen.23547
- **Thermal and mechanical behavior of innovative melt-blown fabrics based on polyamide nanocomposites** ,Dorigato, Brugnara, Giacomelli,Fambri,Pegoretti, Journal of industrial textiles , December 2014.

1. Development of a PA6 polymer and/or yarn with flame retardant properties built-in
2. Development of a PA6 polymer and/or yarn with superior anti staining features

Considerations:

- PA6 does not have flame retardant properties.
- PA6-based carpets normally are not able to pass the tests required for the application in the transport sector (aircraft, ships, trains).
- PA6-based carpets application in the transport is anyhow possible; the solution comes from the carpet manufacturers, who work on the coating composition (back side) by charging along with the latex a lot of inorganic fillers and flame retardant agents.
- This has an impact on the manufacturing cost and recyclability and it is also mostly dependent on the carpet structure and construction.
- Other possibilities are a blend between wool and PA6 fibre, but also in this case there is a negative impact on recyclability.
- Therefore a development of a PA6 yarn with inherent flame retardant properties would represent a real market break-through.

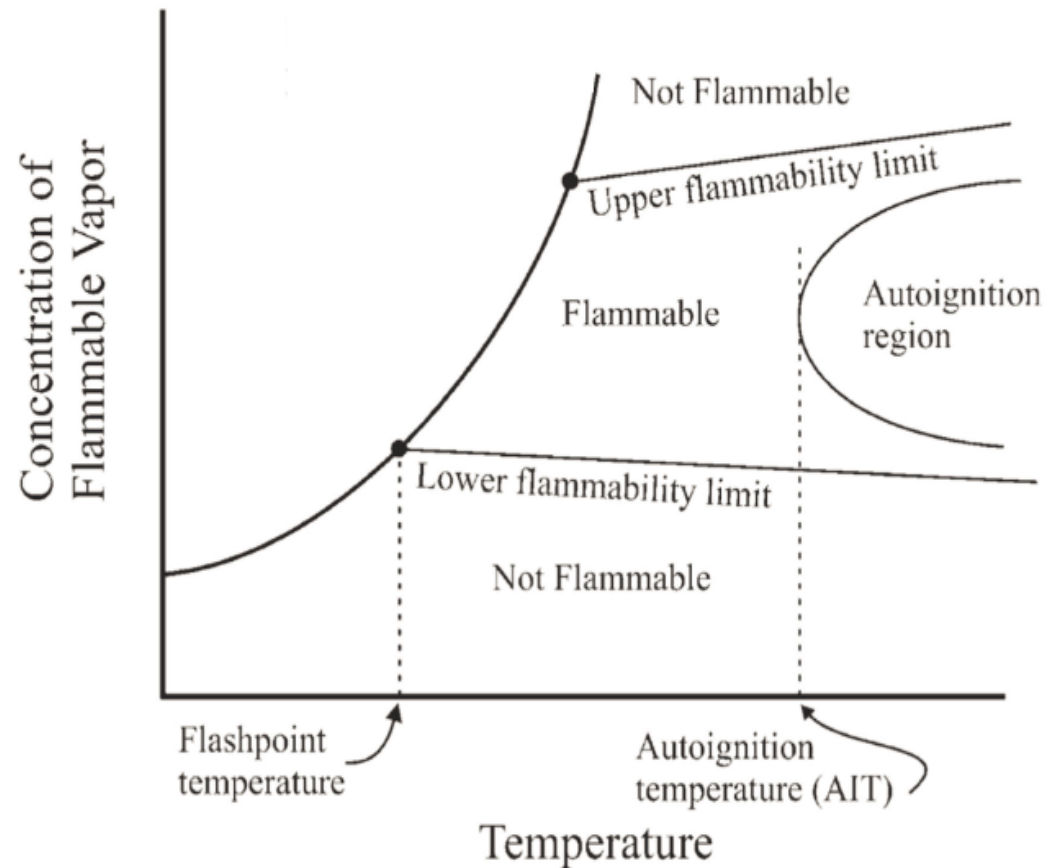
The approach to the research took care of the following aspects:

- Patent status and literature analysis to see what already exists (state of the art)
- Benchmarking analysis (Wool, PET FR)
- Information about legislation and testing procedures
- Study of the reaction mechanism of the main flame retardant additives
- Identification of the all potential additives to be tested commercially available (P based, Melamine based, nanoparticles, metal oxide and their combinations)
- Selection of those with a chemistry compatible to PA6, with no impact on health and safety, and complying with the current legislation (i.e. halogen free)

Flame Retardant Project

Flammability – considerations:

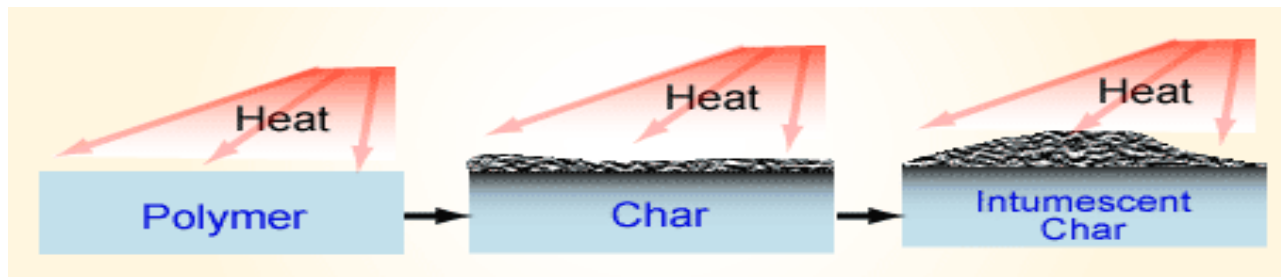
- Only gas material can burn: solid and liquid needs to pass in gas phase first
- A gas mixture can burn only if the combustible concentration is inside a specific interval, between LFL and UFL
- There are two significant temperatures:
 1. Flash point: minimum T at which a solid/liquid can develop a flammable gas mixture
 2. Autoignition T: no need of spark, temperature is enough to cause ignition



Flame retardant products may work either on a chemical way or on a physical way

Examples of additives working with a chemical reaction are:

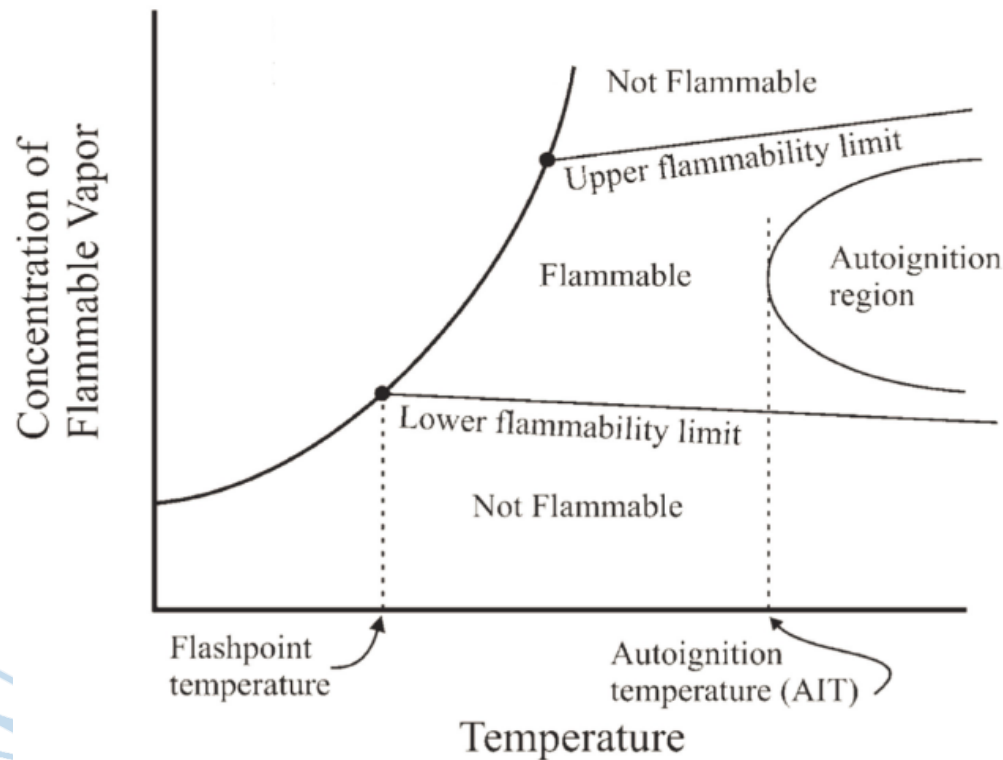
- Halogen based product (radical scavenger) – action in gas phase, making the concentration exit from the flammability region
- Forming an intumescent layer, like the P based – action in solid phase, avoiding the formation of a flammable mixture



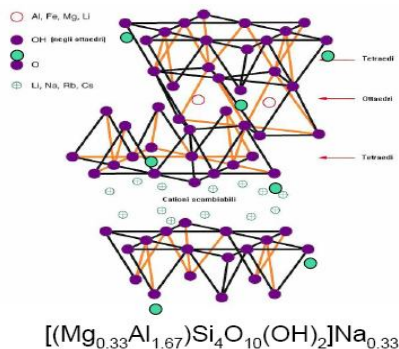
Flame Retardant – Action

Examples of additives working with a physical reaction are:

- $\text{Mg}(\text{OH})_2$ or $\text{Al}(\text{OH})_3$ – water release / endothermic reaction
- Phosphor based additives that form a protective layer



Flame Retardant – Additives



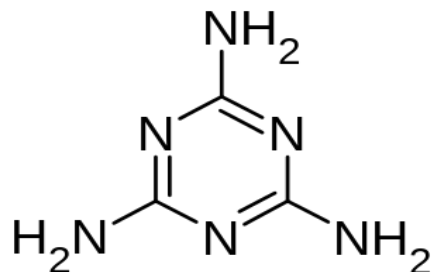
Nanoparticles (clay)



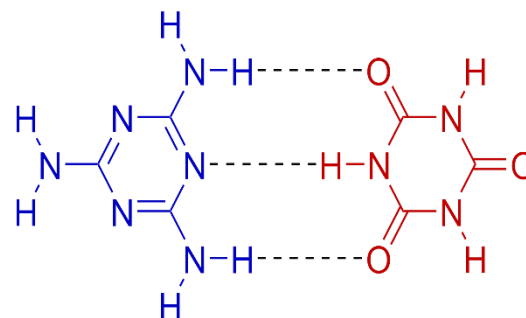
Aluminium hydroxide



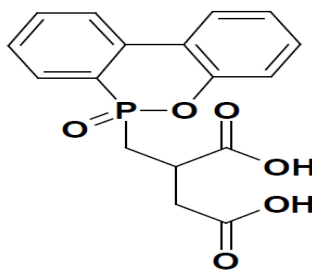
Magnesium hydroxide



Melamine



Melamine cyanurate



Butanedioic acid(6-oxide-6H-dibenzo (c,e)(1,2) oxa-phosphorin-6-yl)

There are different kind of testing procedures, according to the final product in which the yarn is used:

- **Aircraft**
 - AITM 2.0002B (FAR 25853, 12” Vertical Bunsen Burner Test)
 - AITM 2.0007B+3.0005 (smokes density & toxicity)
- **Railways**
 - EN ISO 9239-1 (Radial panel-tilted)
 - ISO 5660-1 (Heat release, cone calorimeter)
 - ISO 5659-2 + IR (smokes density & toxicity)
- **Ships**
 - MSC61(67) Part 5 o IMO res A 653 (Radiant panel-vertical)
 - MSC61(67) Part 2 (smokes density & toxicity)

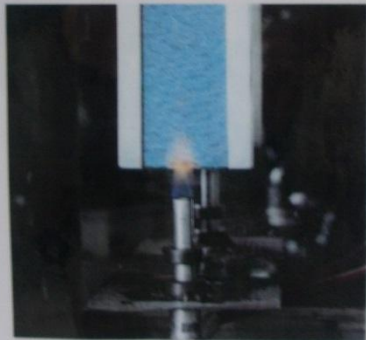
Flame Retardant – Testing

- Vertical Bunsen Burner (aircraft)

12" vertical Bunsen Burner Test

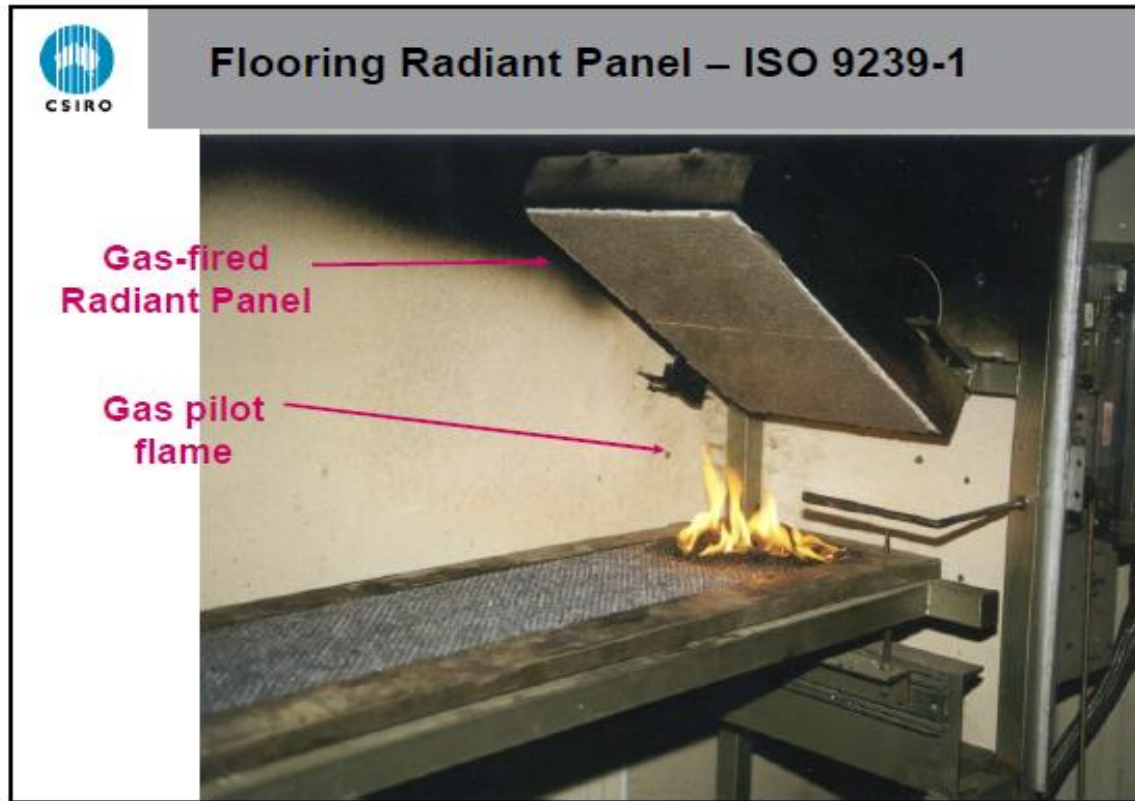
Standard: FAR 25.853, App. F, Part I
Requirements: Max. burn length of 20 cm
Extinguishing time of 15"

Apply time: 12 seconds
Flame temperature: Min. 843 °C
Flame height: 38 mm
Specimen: 305 mm X 76mm
3 in each direction



Flame Retardant – Testing

- Radiant panel (railways)



- To obtain a yarn with FR properties, there are basically 3 ways:
 - ✓ during polymerisation (by modifying the polymer structure)
 - ✓ by addition of a FR additive as a master batch during spinning
 - ✓ in a post treatment during the reprocessing of the yarn (i.e.: topical treatment during heat setting)

- ✓ Project is still going on through polymerisation (polymer modification)
- ✓ The right molecules to be used in the reaction vessel have been identified
- ✓ Process conditions to be applied have been tested
- ✓ The first polymer is already available for testing purpose
- ✓ First results look very promising

Flame Retardant Status

Test UL94 – standard polymer



Test UL94 – FR polymer



Considerations:

- Carpets can be made from all kinds of fibres: polypropylene, polyester, acrylic, silk, wool and nylon.
- Because of its powerful resilience and resistance to abrasion, nylon is preferred for commercial area.
- While no carpet is 100% stain-proof, if the pile is made from a stain-resistant yarn, this makes easier to remove most stains quickly and without permanent damage.
- Therefore the development of an enhanced anti-stain nylon 6 fibre will be a plus and an important reason for the consolidation and expansion of the market position.

- Textile floorcovering need to be regularly maintained and cleaned whenever a spills may occur



- It is important to clean the carpet surface soon after the spill



Stain formation is similar to dyeing and painting

- stains inside and outside the fiber, with molecular attraction and diffusion of the staining elements inside the material (disperse type staining): wine, coffee,
- stains inside the fiber, involving a molecular attraction, diffusion and a chemical reaction between the material and the staining material (acid staining): beverages, ...
- surface stains, where the staining substance is spilled out onto the surface or material and is trapped in the fibers, pores, indentations or other capillary structure of that surface (mechanical adhesion): shoes polish, ...

Staining

- The reason why foods and beverages are staining is that they do contain specific organic colorants, natural or synthetic, i.e. organic molecules that can diffuse and stick to the carpet
- Staining is a mechanical, physical and chemical (chemical reaction) adhesion of the colored part of the stains on and within the carpet pile.

Acid staining

- Artificial colorant are often added to the foods to improve their appeal to the public and are also used in shoe polish, lips sticks, ..
- Those artificial colorant are in fact real dyestuffs, i.e. organic pigments able to penetrate inside the polymer
- Some are acid dyestuffs, i.e. they can chemically link to the acid dye able nylon (e.g.: E129 = Red40).

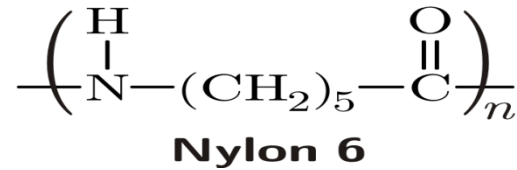
Anti Staining project



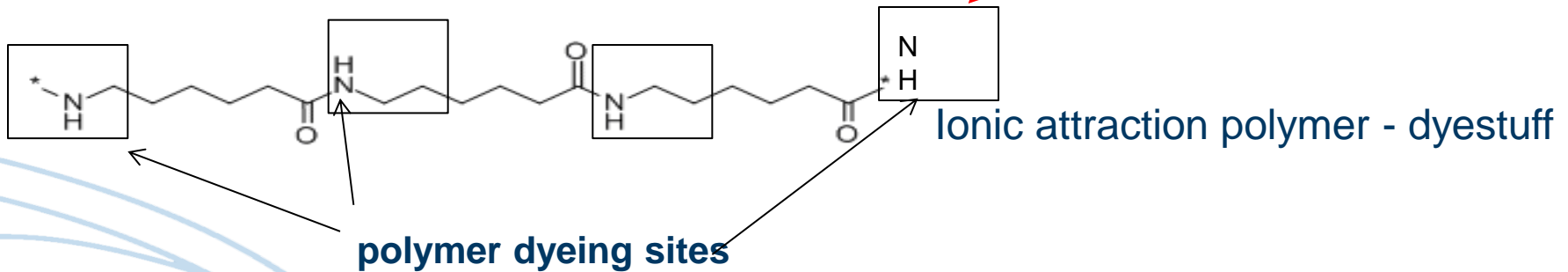
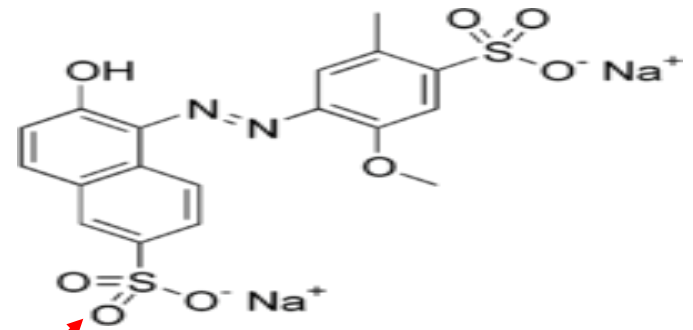
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Nylon 6 polymer acid staining

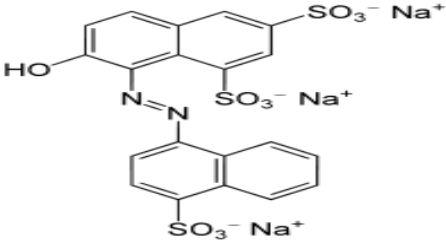
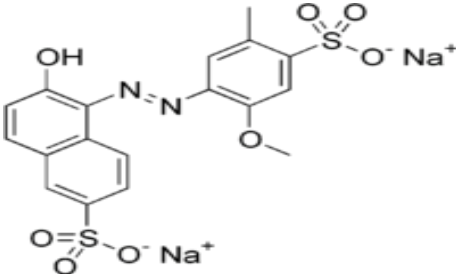
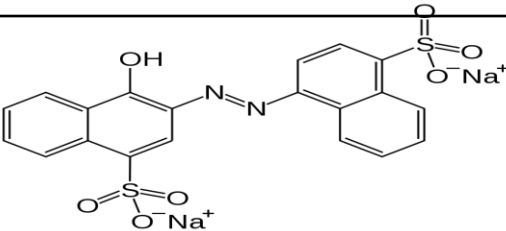
Regular Nylon 6

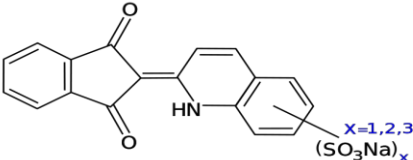
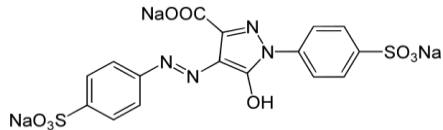
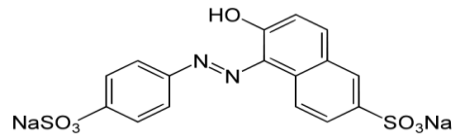
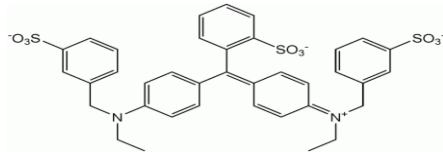


Acid dyestuff



Anti Staining - Chemistry

	Dyestuff		
	Name	Color	Chemical structure
E124	Ponceau 4R	Red	
E129	Allura AC Red40	Red	
E122	Azorubina	Red	

	Dyestuff		
	Name	Color	Chemical structure
E104	Yellow Chinolina	Yellow-green	
E102	Tartrazine	Yellow	
E110	Yellow FCF	Yellow	
E133	Blu FCF	Blu	

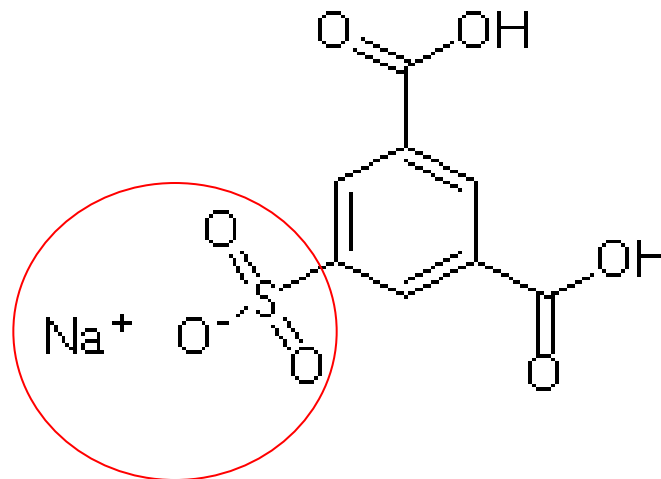
Acid Stain Resistant carpets

Two options to improve acid stain resistance of nylon :

1. use a stain-blocking system
 2. use modified polymer
-
1. Acid dye blockers or stain blockers are resins (negatively charged anionic aromatic compounds) that act like colorless dyes: by occupying dye sites, they make nylon fibers to resist acid dyes. They are added to the nylon by the fiber producer or by the carpet mill and they are not permanent.

Acid Stain Resistant carpets

2. A modified nylon 6 polymer is produced by introducing in the polymer chain some molecules that contain a sulfonic groups

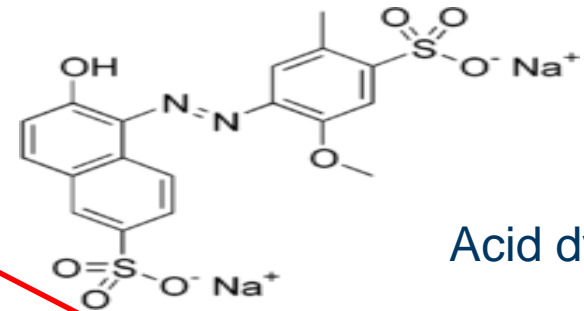


Acid Stain Resistant nylon

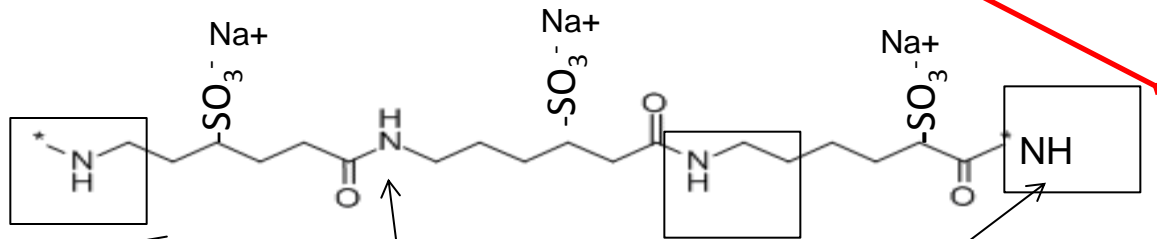
- The introduction of sulfonic groups (or other negatively charged molecules) leads to physical repulsion of the acid dyestuffs.
- This is the base of the use of the cationic polymer in most important nylon fibers (several patents issued on this)
- This is important to limit the range of possible stains (moreover, in some cases, also coffee and wine can have an acid behavior)

Modified cationic nylon 6 polymer

Ionic repulsion between
polymer dyestuff



Acid dyestuff



polymer dyeing sites
'protected' by SO₃⁻

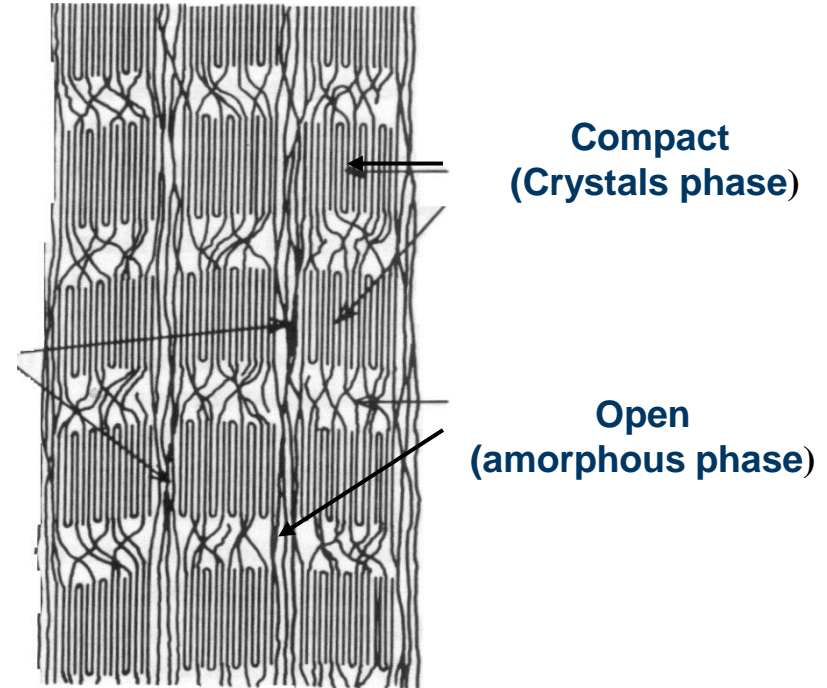
Cationic Nylon 6

To enhance the resistance of a PA6 carpet to acid staining, the introduction of cationic groups is an essential but not sufficient condition.

- Results depend not only from the presence of $\text{SO}_3^- \text{Na}^+$ groups but from their amount (ppm or % added), from their position in the polymer chain (chain ending or intra chain) and from the ratio Na/S.
- Results are also depending on the number of the dyeing sites available on the polymer (NH_2 groups)
- Capability of the dyestuff to get access to the dyeing sites which is related also to the yarn crystallinity level (physical mechanism).

Stains penetrate inside the yarn through the 'channels' of the open part structure, then react with nylon dye sites -NH_2 .

For water-based stains (wine, tea, coffee, beverage, ...) hydrophilicity play an important role.

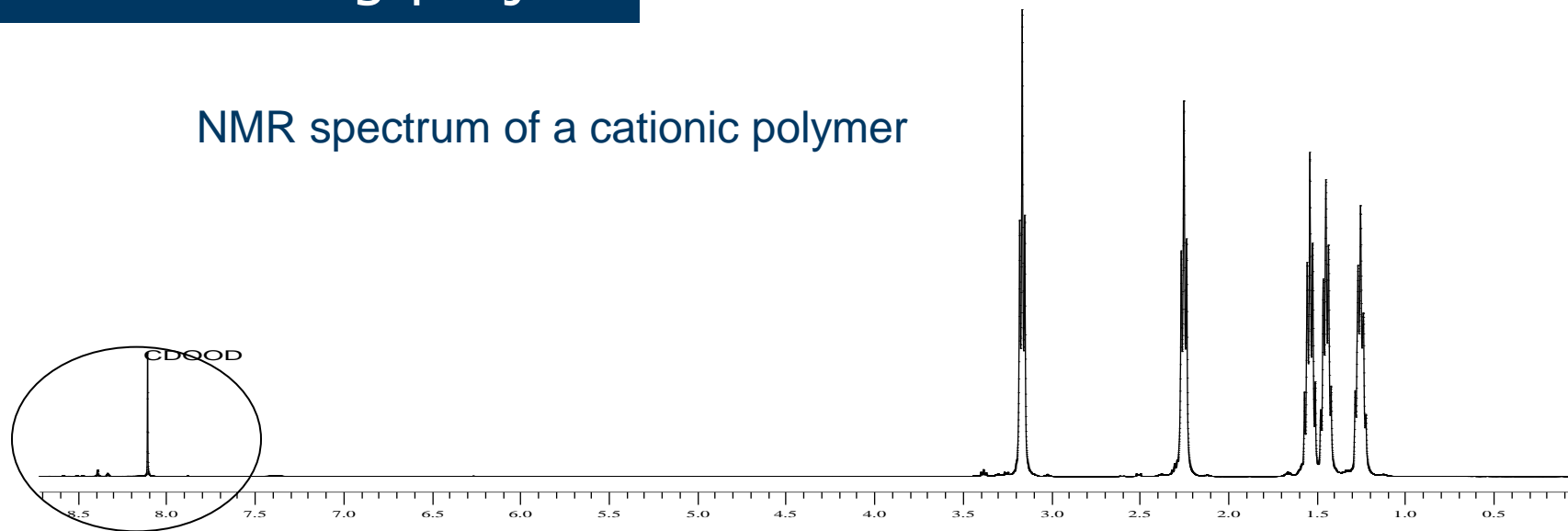


**Structure of synthetic filaments
at high magnification**

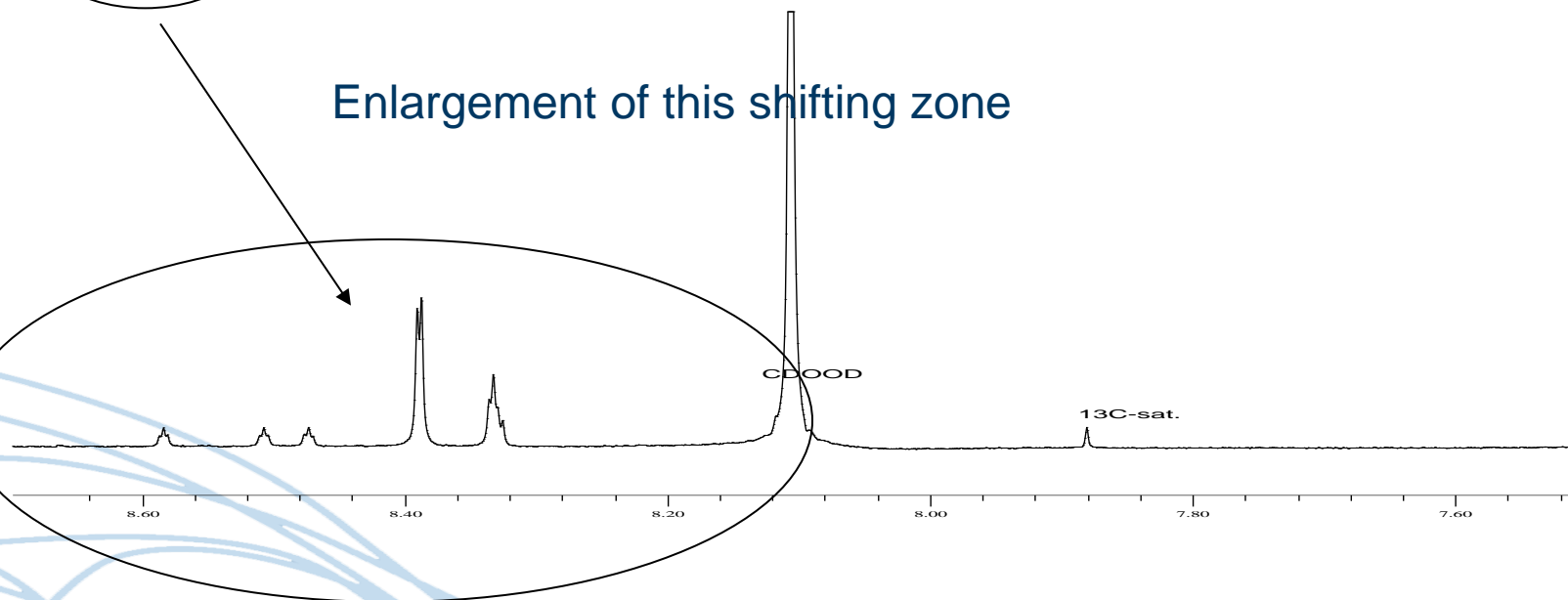
- All these factors together (both chemical and physical) play an outstanding role in granting the anti-stain resistance of the carpet
- At a polymer level, it is important to know as much as possible about SO_3^- groups, their position and level.
- Therefore, in collaboration with a Dutch University, it was developed a lab method, through NMR, for the determination of the amount of the $\text{SO}_3\text{-Na}^+$ groups in the polymer chain and their distribution in the chain (end-chain, inter-chain and free)

Anti Staining project

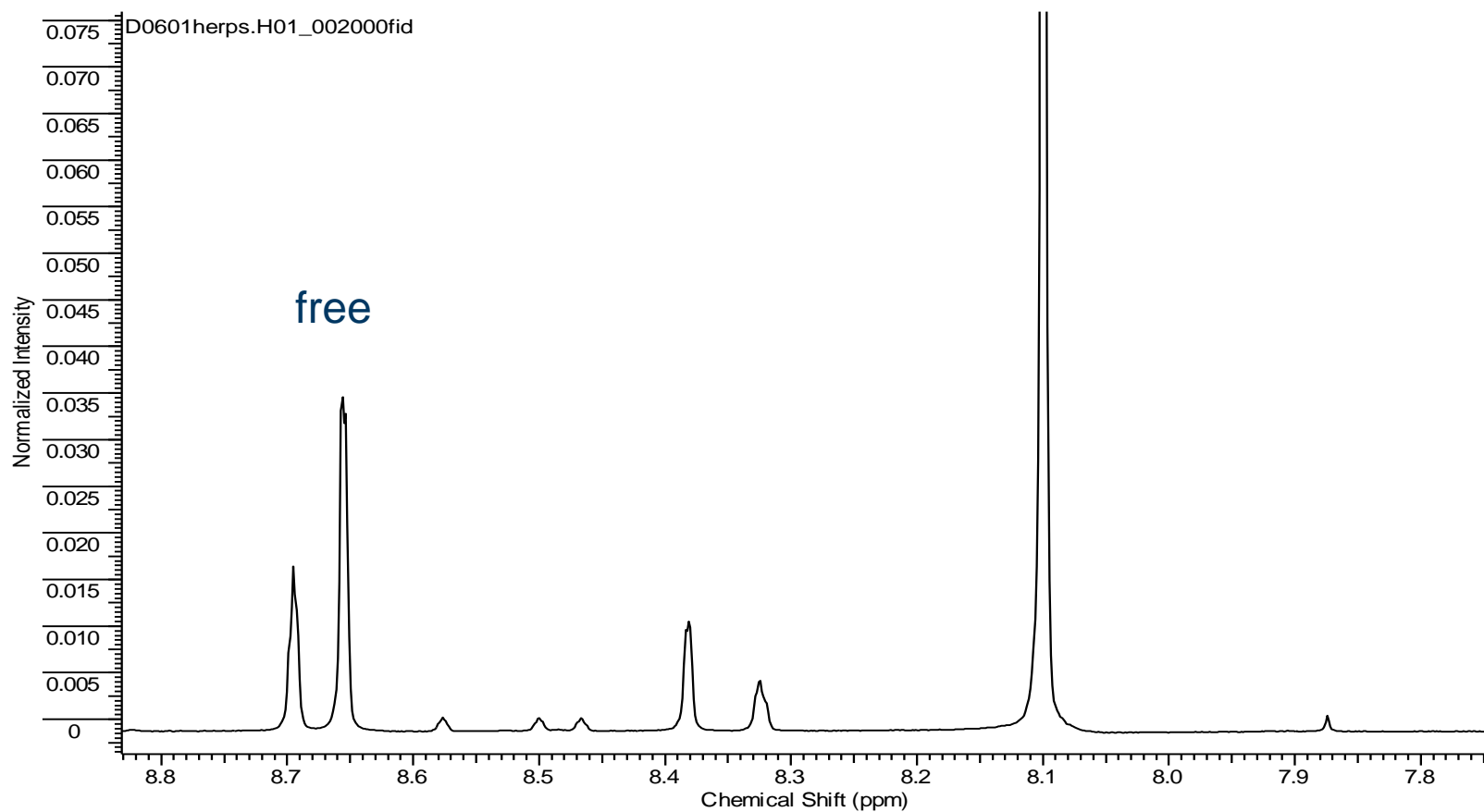
NMR spectrum of a cationic polymer



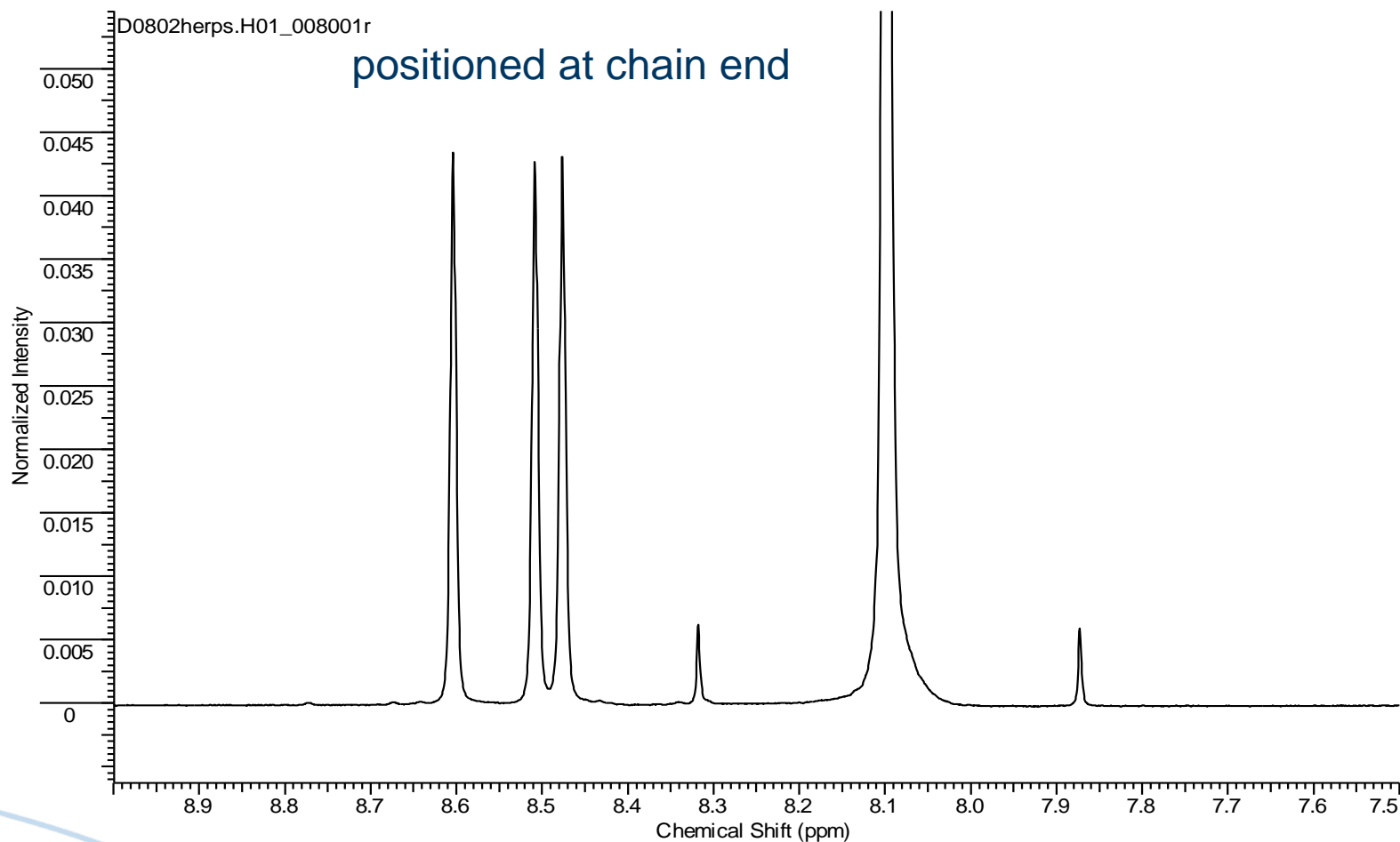
Enlargement of this shifting zone



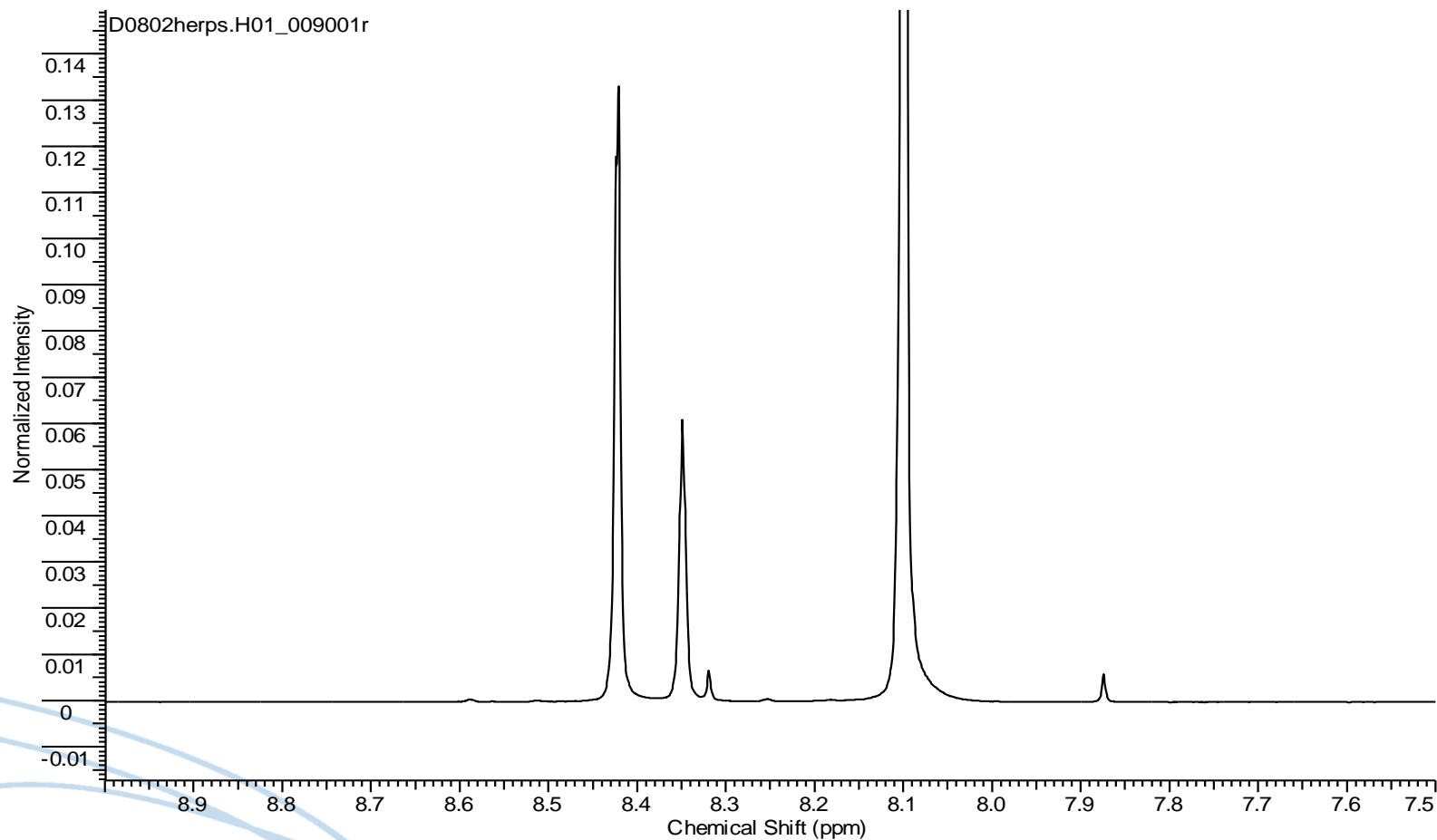
Anti Staining project



Anti Staining project



inter chain positioned



Cleaning results of an Acid Stain Resistant carpet



Regular
PA6

Modified
PA6

Cleaned after 24 h

Cleaned soon

Stained

Technology Transfer & Production Upgrade

Technology transfer activity plays an outstanding role for the BCF business

Since the manufacturing sites are located on the three continents , the goal is to ensure that process & product improvement are always implemented at the same level and with the same efficiency

Manufacturing steps typically involved are:

- Polymerisation (Polymer Production)
- Coloured Master Batch Production
- Extrusion (BCF spinning, yarn manufacturing)
- Yarn Reprocessing (AE, TW, HS-SS, HS-SU)

Targets to be achieved might be the most different ones, such as upgrading of an existing product, development of a new yarn range, or implementation of new technological process conditions.

Technology Transfer & Production Upgrade

Like other business areas also for the textile floorcovering business there are several drivers pushing towards a continuous product innovation with increased performances.

The main factors that always push and drive product & process upgrade are:

- **Improvement of the internal efficiency** (process yield, costs reduction, alternative raw materials, additives or pigments sources, etc)
- **Improvement of the product (carpet) performances** (superior bulk/pile cover or finer count (dtex) but ensuring the same wearing resistance)
- New market needs (**product functionality** like carpets of easy maintenance and easy cleanability)
- **Refreshing the market appealing** (launch of new colour range)
- **Fighting against the main competitors** to keep the market share

Technology Transfer & Production Upgrade

As example we take the **development of a new solution dyed yarn for automotive**



We serve all the major cars manufacturer (BMW, Audi, Mercedes, Ford , Chrysler , Peugeot) providing them the same product for the same car model, from Arco for the EU market, from Cartersville for USA and from Jaixing for the Asian market.

Therefore once a new development is approved and definitely launched, the product must be the same worldwide in terms of textile features and colour features.

In the automotive production chain two main steps are involved:

- preparation of the coloured masterbatch
- yarn extrusion

Color Masterbatch

Masterbatch is a mixture of polyamide, organic and/or inorganic coloured pigments, heat stabilizers and UV stabilizers, used to:

- give the yarn a colour shade while it is extruded
- ensure the yarn the needed light and heat fastness to withstand the severe automotive testing procedure
- enhance the mechanical strength to enable the yarn to undergo the thermal / mechanical stress of the carpet moulding

Color Masterbatch preparation

Polyamide 6

EXTRUSION

Color Pigment

**Monoconcentrated
Masterbatch
(20 – 50 wt%)**

EXTRUSION

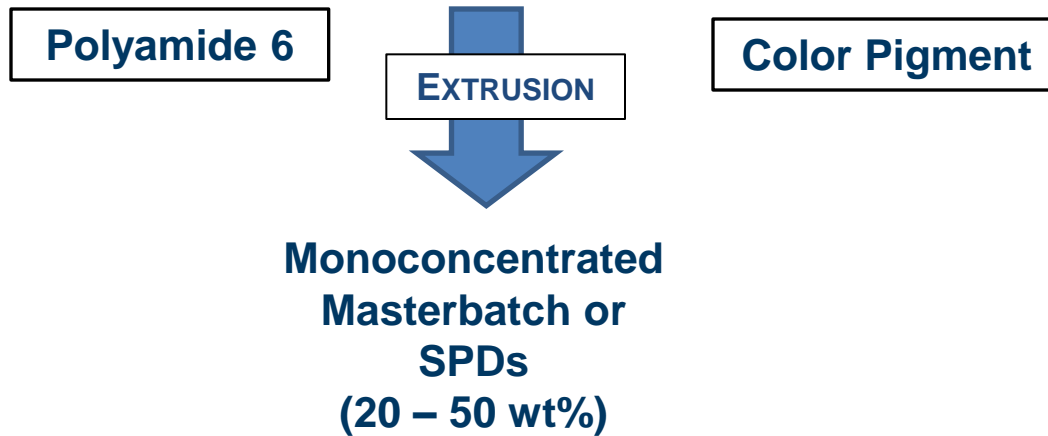
**Color
Masterbatch
(10 – 25 wt%)**

MELT SPINNING

**Melt Spinning Fibers
(0.3 – 1 wt%)**



Color Masterbatch preparation



- Production of SPD (single pigment dispersion) is made in Arco site only
- SPDs are then used to prepare the colored masterbatches necessary to manufacture the colored yarn
- A good, reliable and consistent color reproduction mostly depend on the SPD's quality (pigment dispersion level)
- Therefore batch to batch consistency is regularly checked to ensure that dispersion level is always the same
- Filter test and color strength are then measured whenever a new campaign of SPDs is manufactured

Color Masterbatch preparation

Main inorganic pigments

Pigment	molecule	CAS number	color	chemical formula	MW	density [g/cm ³]	dimension [nm]
WHITE PW6	TiO ₂	13463-67-7	white	TiO ₂	79,90	4,2	300
BROWN PR101	iron oxide α-Fe ₂ O ₃	1309-37-1	brown	Fe ₂ O ₃	159,69	5,2	90
YELLOW PBr24	Chrome antimony titanium buff rutile	68186-90-3	yellow	(Ti,Cr,Sb)O ₂		4,5	688
BLACK PB7	Carbon black		black	C			



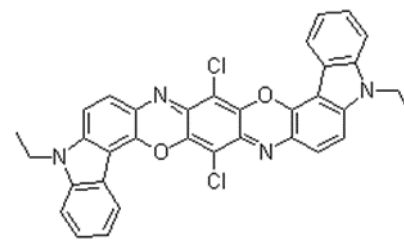
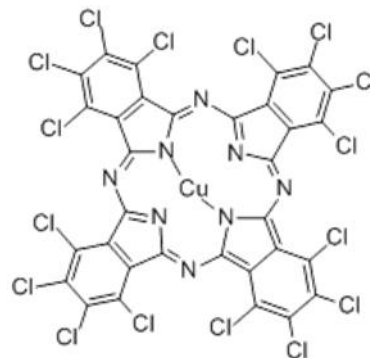
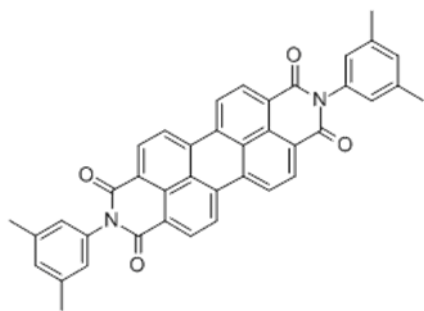
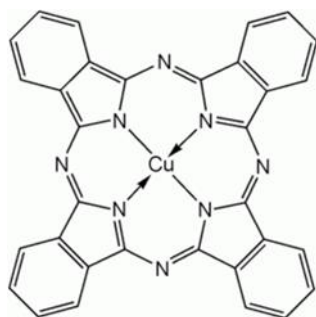
Features:

- high absorption of light and small scattering, except for TiO₂
- stable at high temperature above 300° C
- lower color strength compared to organic pigments
- good dispersion capability even at low shear stress
- good wettability of powders

Color Masterbatch preparation

Main organic pigments

Pigment	molecule	CAS number	color	chemical formula	MW	density [g/cm ³]	Dimension [nm]
BLUE PB 15:3	Cu-phthalocyanine beta	147-14-8	blue	C ₃₂ H ₁₆ CuN ₈	576,07	1,6	220,8
RED PR149	Perylene	4948-15-6	red	C ₄₀ H ₂₆ N ₂ O ₄	598,66	1,4	125
GREEN PG7	Cu-phthalocyanine halogenated	1328-53-6	green	C ₃₂ H ₃ N ₈ Cl ₁₅ Cu	1094,77	2,1	201
VIOLET PV29	Dioxazine	6358-30-1	violet	C ₃₄ H ₂₂ Cl ₂ N ₄ O ₂	589,48	1,5	

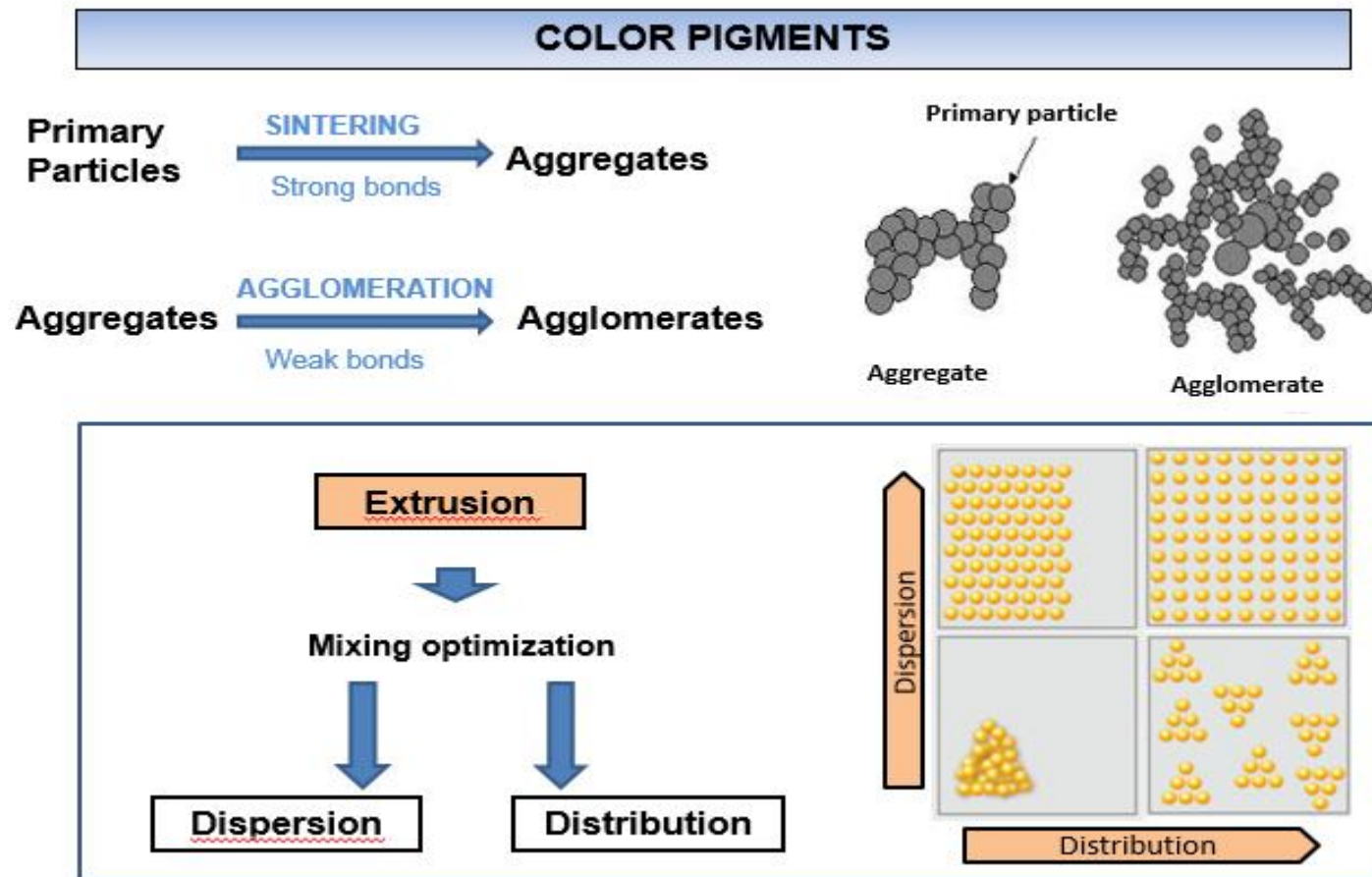


Features:

- polycyclic types containing chromophores groups
- insoluble
- poor dispersing ability
- heat resistance often limited to about 280° C
- at high temperature the chemical interaction can lead to irreversible changes in pigment structure
- possible reduction effect on amine groups of polyamide

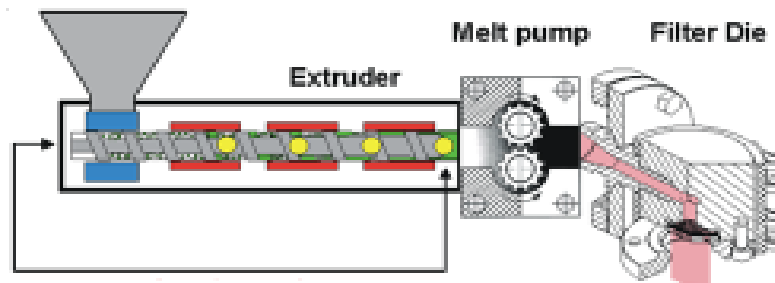
Color Masterbatch preparation

Pigments dispersion and distribution



Testing procedure to assess the pigments dispersion

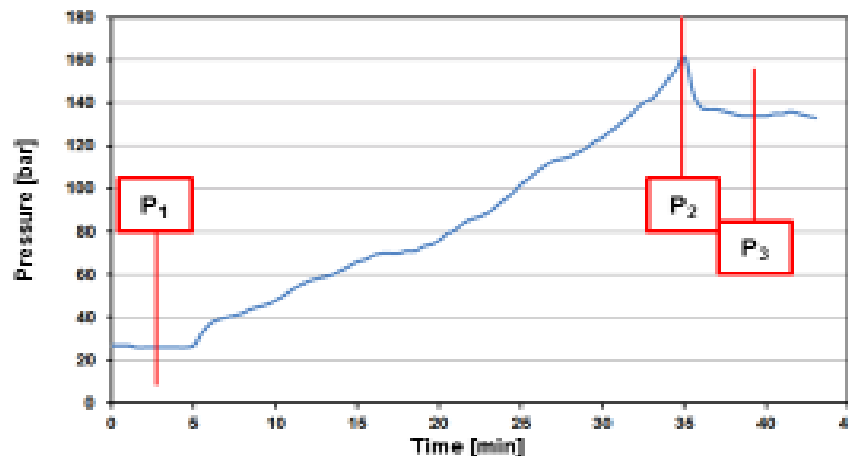
EXPERIMENTAL TECHNIQUES - FILTER-TEST



- Indirect method
- Pigments dispersion
- Representative of the product
- Repeatability of analysis

FPV = Filter Pressure Value [bar/g]

Referred to DIN EN 13900-5



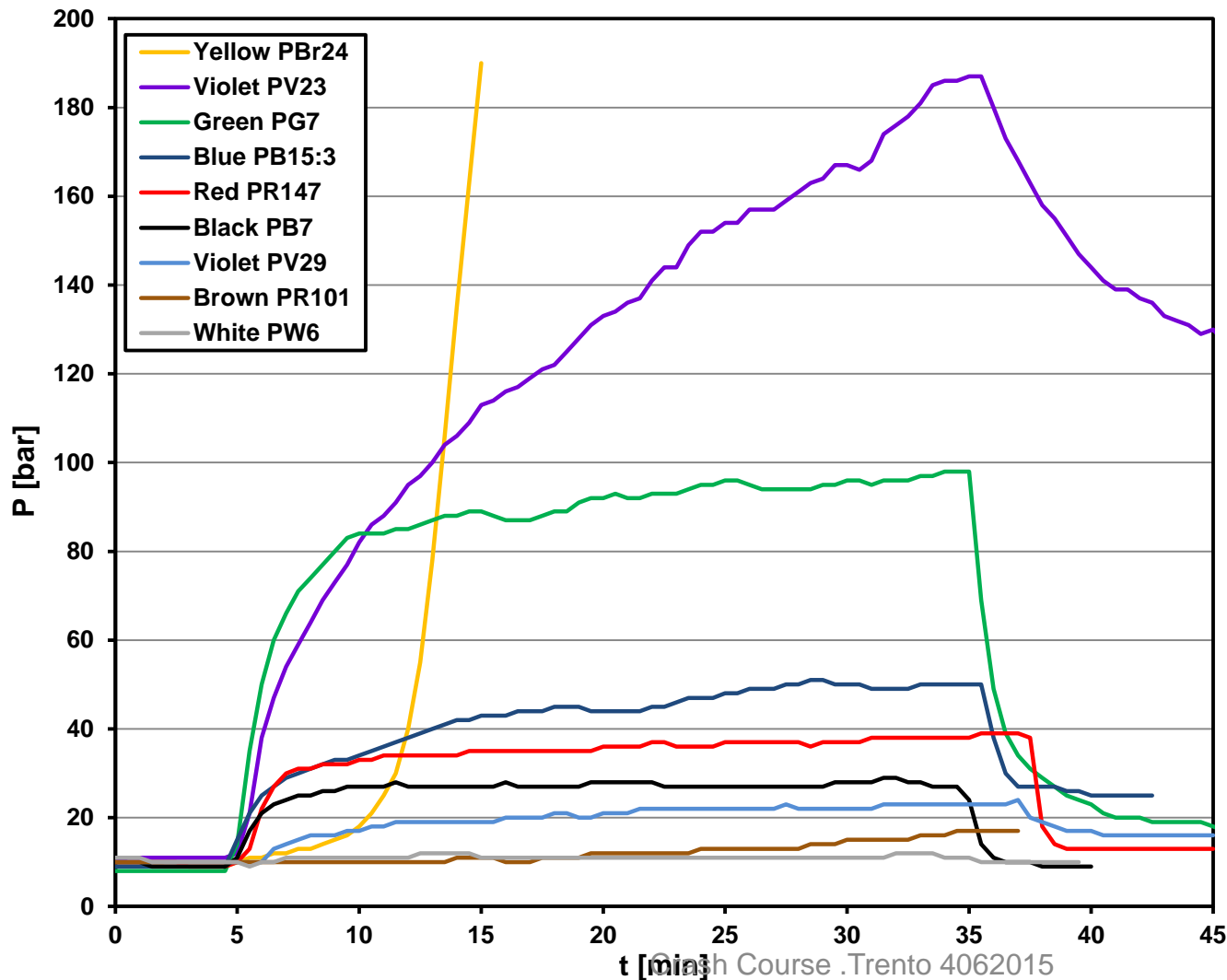
$$FPV = \frac{P_2 - P_1}{M} \left[\frac{bar}{g} \right]$$

$$FPVII = \frac{P_3 - P_1}{M} \left[\frac{bar}{g} \right]$$

P1 Initial Pressure (1st Base line)
P2 Peak Pressure
P3 Final Pressure (2nd Base line)
M Mass of pigment

Color Masterbatch preparation

Critic pigment selection → Filter-Test - 25 µm Filter Sintered



Dilution: 25%

Organic pigments

Violet PV23
Green PG7
Blue PB15:3
Red PR147
Violet PV29

Inorganic pigments

Yellow PBr24
Black PB7
Brown PR101
White PW6

Color Masterbatch preparation

Color strength Determination

Spectrophotometer



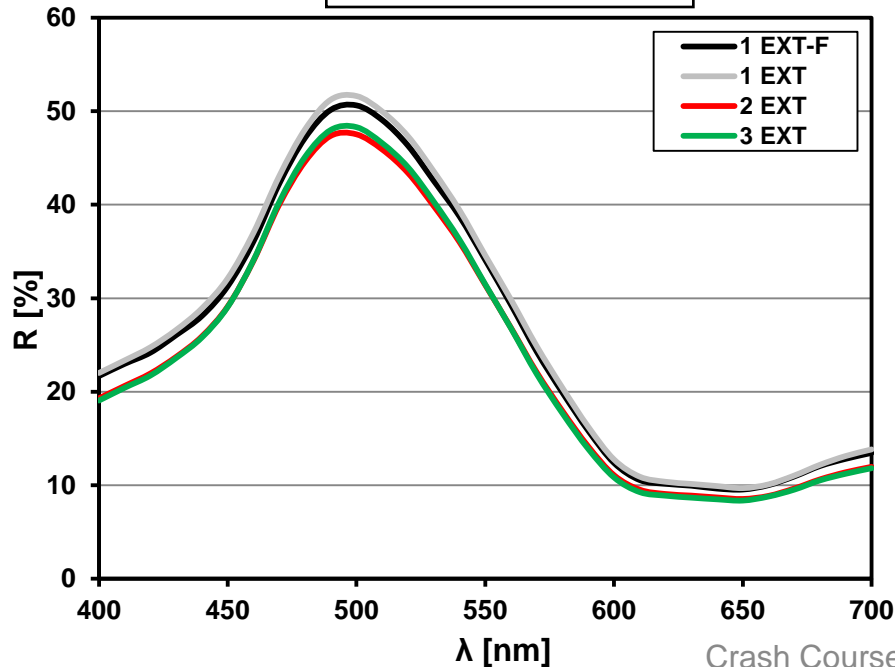
Reflectance Curve

$$RCS = \frac{(K/S)_{sample}}{(K/S)_{reference}} \cdot 100$$

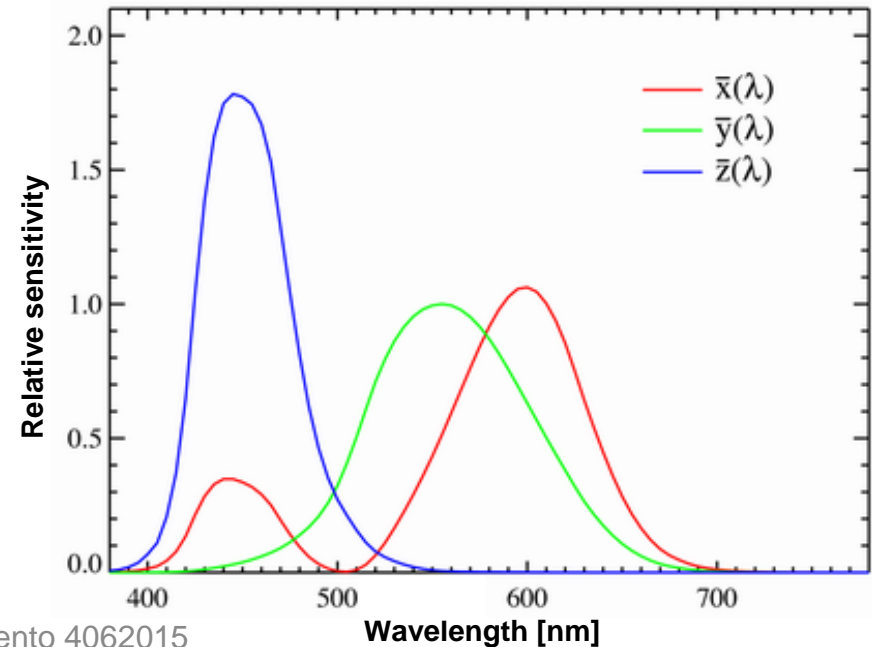
Relative Color Strength

Where: - K is absorption coefficient
- S is scattering coefficient

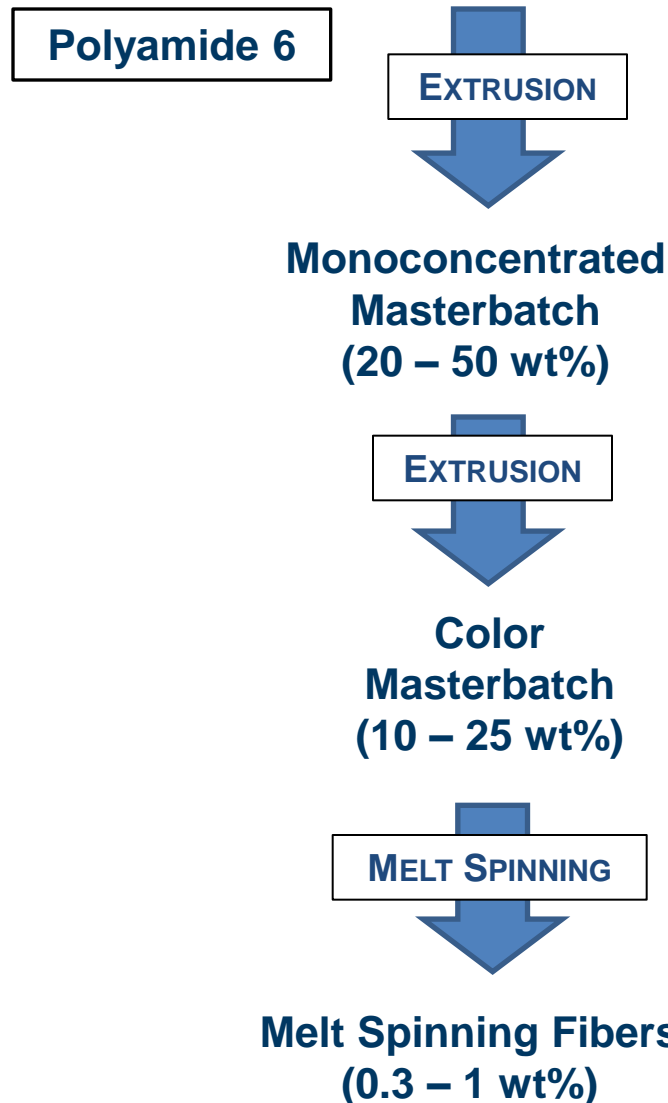
Reflectance Curves



Color matching functions



Color Masterbatch preparation



Color Pigment

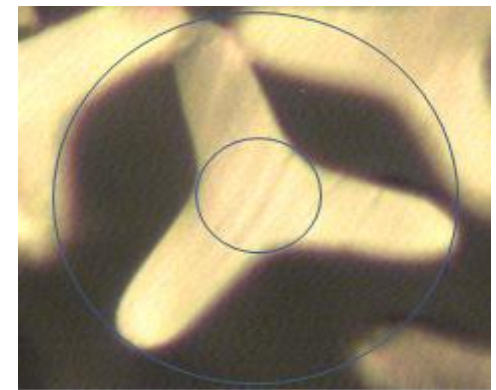
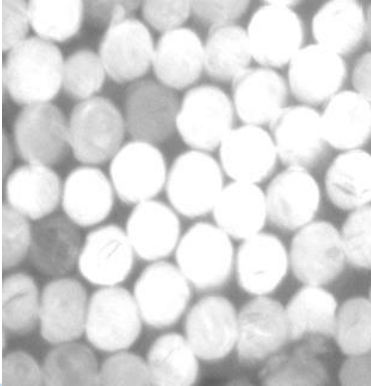
- Colour matching is made using SPDs made in Arco to formulate a new recipe to match the colour shade/ strength of the sample to be reproduced
- If necessary correction to Masterbatch recipe is done if colour shifting is present on yarn
- This activity takes place in all the three production sites (Arco, Cartersville, Jaixing)

Steps for the validation of new colour

- Colour matching is made using SPDs to formulate a new recipe to match the colour shade/ strength of the sample to be reproduced
- First yarn sample extrusion to check if the colour strength is achieved
- If necessary correction to Masterbatch recipe is done if colour shifting
- Then the colour fastness is assessed through Xenotest according to Fakra conditions (4 cycles , BP Temperature 90° C)
- Then mechanical features are tested (160° C for several hours, 50% retained original tenacity / elongation)

For the development of a new yarn for automotive, the steps are the following:

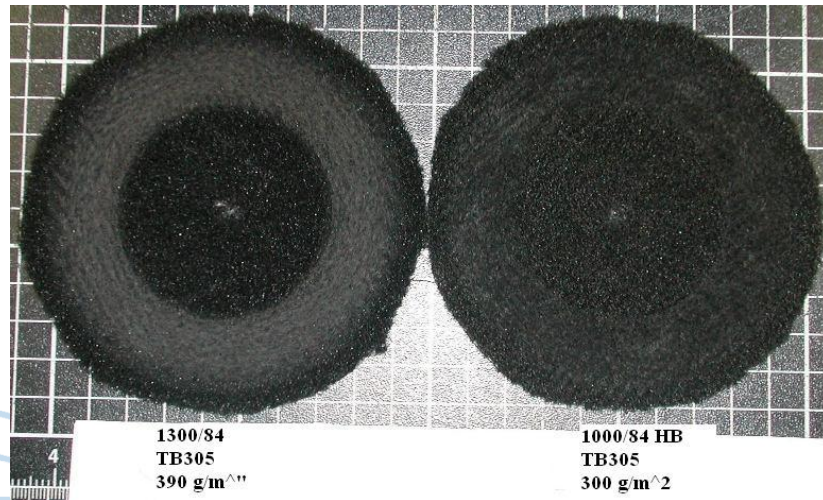
- Setting the targets to be achieved, like principal yarn features (yarn count - dtex-, filament number -d.p.f.- , texture level -bulk-, cross section shape , mechanical features, colour fastness to light & heat, etc)
- Study of the spinnerets capillary size & dimension to get a good spinnability (shear rate) and the desired size and filaments shape



- Setting the extrusion/spinning, drawing & texturing conditions to run with

- Production of the first samples (might be pilot plant or directly on small production line)
- Product full characterisation (textile features, chemical features, wearing test (Taber), light and colour fastness (Xeno-Fakra), carpet evaluation)

Picture of the Taber test



Colour fastness



- Sample is then submitted to the customer interested and then feedback is waited
- After positive feedback, the following step is the industrialisation phase, moving the yarn from pilot plant to industrial line
- **This is a very delicate phase since pilot plant setting is not completely fitting to the industrial one (machine might differ in quenching zone, spinning height, spinnerets size, texturing unit, cooling drum vacuum, winder)**
- Therefore this technology transfer phase is of extreme importance
- It is always supported (as physical presence in situ) from a process engineer who deals with all the details in order to have a very smooth switch .

Aquafil S.p.A. Synthetic Fibres and Polymers

Arco, via Linfano 9

www.aquafil.com

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