# Control the process to control the product: The use of Ludovic Software for optimizing extrusion compounding process parameters





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# Let me introduce myself



## **Research** activities

- Evaluation of the <u>property/structure relationships</u> of polymeric materials, including innovative materials from renewable or recycled sources.
- <u>Valorization of agro-industrial waste</u> for the production of high-performance biocomposites.
- Development of <u>innovative formulations for blends and composites</u> tailored for various applications using different processing technologies (twin-screw extrusion, injection molding, compression molding, 3D printing, cast extrusion).
- Formulation of <u>bioactive coatings</u> for surface treatments or edible coatings for fruits and vegetables to ensure specific properties on substrates made of different materials.
- <u>Optimization of process parameters</u> for primary and secondary polymer transformation operations to enhance the thermo-mechanical properties of the final products.
- <u>Testing</u> of the mechanical, technological, and thermal properties of polymers and composites following standard or customized procedures for the final application. Particular attention is dedicated to mechanical properties, interphase adhesion, plasticity, creep, fatigue, and fracture mechanics.



## Welcome!!









# "

## More than a substance, plastic is the actual idea of infinite transformation

"

**Roland Barthes** 

## How to pass from polymer synthesis...

Polymers are made up of macromolecules, i.e. molecules that are flexible and enormously long compared to the size of the individual monomers that make them up.



## ...to achieve good plastics?





*Plastics* as technical materials are based on polymers (or macromolecular substances), but in most cases they contain a number of added components. Such an added material may be another polymer; in this case we have a polymer *blend*. Moreover, there is a large variety of *additives* and *fillers*, compounded into the polymer for various purposes, which are roughly categorized below:

- in behalf of processing:
  - lubricants for the transportation in the processing machine,
  - antioxydants for protection of the polymer against oxydative degradation during processing,
  - sulphur, for the vulcanization of rubbers,
  - accelerators, for speeding up the network forming reaction with rubbers and thermosets,
  - blowing agents, for producing foams,
  - etc.
- in behalf of mechanical properties:
  - plasticizers, e.g. in PVC, to obtain flexibility,
  - quartz powder, mica, talcum etc. to improve the stiffness.
  - short glass fibres, to improve stiffness and strength,
  - rubber particles, to improve impact strength,
  - etc.
- in behalf of other properties:
  - ultraviolet stabilizers, to protect the polymer against degradation in sunlight,
  - antioxydants, for protection against degradation during use at elevated temperatures,
  - antistatic agents, to reduce electrostatic charging,
  - pigments,
  - cheap fillers such as wood flour, for price reduction,
  - flame retarding additives, etc

## **Thermoplastics Polymer Processing**

- Polymer processing is defined as the "engineering activity concerned with operations carried out on polymeric materials or plastic systems to increase their utility".
- Primarily, it deals with the conversion of raw polymeric materials into finished products, involving not only shaping but also compounding and chemical reactions leading to macromolecular modifications and morphology stabilization, and thus, "value-added" structures.
- The conceptual breakdown is that to obtain a plastic item, the base polymer(s) undergo two thermomechanical experiences:
  - 1) Compounding, Blending or Reactive Processing
  - 2) Structuring and Shaping
- The products of the first bullet point are value added and microstructured pellets, while the second is used primarily for fabricating finished products.
  - The important elementary steps for each experience, and the physical mechanisms that affect them, are different, because of the different objectives in each.





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## **Polymer Processing – Elementary steps**



- Subsequent to an operation involving solids handling, the polymer must be melted or heat softened and mixed with all the necessary additives.
- Often this is the slowest, and hence the ratedetermining step in polymer processing, severe limitations are imposed on attainable melting rates by the thermal and physical properties of the polymers, in particular, the low thermal conductivity and thermal degradation.
- The former limits the rate of heat transfer, and the latter places rather low upper bounds on the temperature and time the polymer can be exposed.
- Typically, these operation must be achieved in EXTRUDERS

Polymer

blends

pellets

То

fabricator

## **Polymer Processing - Structuring**

- The final objective in the field of polymer processing is undoubtedly <u>shaping polymer products</u>.
- <u>The selection of the shaping method</u> is dictated by product geometries and sometimes, when alternative shaping methods are available, by economic considerations.
- The molten stream of polymers flowing through dies or into cold molds and it is rapidly cooled to form the solid-product shape.
- As a consequence of the rapid cooling, the retained orientations in plastic products impart specific anisotropic properties to the product and, in the case of crystallizable polymers, special property-affecting morphologies.
- The ability to affect the final product properties is called structuring, which involves many disciplines, and it can be designed to impart extraordinarily different and beneficial properties to plastic products.

(bags, gaylords, RR cars)





## **PLASTIC MOLDING PROCESSES**



## **Current polymer processing practice**

 The elementary steps, as well as the shaping operations, are firmly based on the principles of transport phenomena, fluid & solid mechanics, heat & mass transfer, polymer melt rheology, and mixing principles. These principles provide the basic tools for quantitatively analyzing polymer processing (obviously having basics of physics and chemistry of polymers).



- Recently, to the "classical" polymer processing, engineering analysis and process simulation have been introduced and optimized.
- Many improvements and new developments have led to today's diverse arsenal of sophisticated polymer processing machines, methods and polymer systems of ever-increasing complexity and variety.
- The future of the field is pushed by the demands of predicting, a priori, the final properties of processed plastics or polymer-based materials via simulation, based on first molecular principles and multiscale examination.

## **Polymer rheology**







#### What is viscosity ?

#### Internal friction

Between the molecules and particles, when gliding along each other in a flowing state,



A measure of a fluid's resistance to flow by an applied deformation force.



## **Flow Behavior**

• The shear rate ( $\gamma$ ) - shear stress ( $\tau$ ) relationship can be described by the general equation:

 $\dot{\gamma} = f(\tau)$ 

 Graphically this relation is a curve of shear stress as a function of shear rate. The four basic types of time-independent fluids are shown in the figures below.

- It must be emphazised that these types are an idealization of the real flow behavior of fluids.
- Most polymer solutions and melts exhibit shear thinning. They belong to the class of pseudoplastic materials,
- For polymer melts, shear-thickening or dilatant behavior is rarely observed.



## **Polymers: Shear thinning behavior**

- Shear thinning occurs because of rearrangements in the fluid microstructure in the plane of the applied shear.
- It is frequently seen in dispersions such as suspensions and emulsions, including melts and polymers.



Molecules are coiled, entanglements

Deformation in shear direction, disentanglements

#### Polymers

With chain like macromolecules

## Focus on Polymers: shear thinning behavior

- The observed **shear thinning** of polymer melts and solutions is caused by disentanglement of polymer chains during flow.
- Polymers with a sufficiently high molecular weight are always entangled (like spaghetti) and randomly oriented when at rest. When sheared, however, they begin to disentangle and to align which causes the **viscosity to drop**.
- The degree of disentanglement will depend on the shear rate. At sufficiently high shear rates the polymers will be completely disentangled and fully aligned. In this regime, the viscosity of the polymer melt or solution will be independent of the shear rate, **i.e. the polymer will behave like a Newtonian liquid again**.
- The same behavior is true for very low shear rates; The viscosity at infinite slow shear is called zero shear rate viscosity ( $\eta_0$ ).



## **Processing: Typical values of shear rate**



## Primary Processing, a typical example

In general, the **Extrusion** is a manufacturing process by which a heated material is pushed through a die of the desired cross section.



- In the plastics industry, the diameters of twin-screw extruder screws range from 12 mm to over 400 mm, with production capacities ranging from 0.250 kg/h to over 50,000 kg/h.
- Segmented screws convey and shear the materials processed in channels delimited by screw flights and barrel walls, with short mass transfer distances.

#### **EXTRUDERS FOR THERMOPLASTICS**



## SINGLE SCREW EXTRUDER

- On a single screw extruder one screw rotates in the barrel. It is possible to obtain different geometries so it can be used in different applications. In particular, it is mostly used for processing thermoplastic into semi finished products.
- Pressure generation and material conveying are the most important functions of this kind of extruders. This, single screw extruders usually have an increasing diameter from the hoppers to the die, in order to generate greater pressure. Because of having only one screw, the mixing is very poor consequently they cannot be used for extrusion compounding.



## **Our interest: Twin-screw extrusion**

It is the main technique to achieve the **extrusion compounding process**, which implies the addition of **fillers**, **plasticizers** and other additives to a polymeric matrix to achieve a material which meets the **final requirements** for every application. The goal of this lesson hence is to make a broad description of the extrusion process and the main parts of the machine.





#### Screws





#### **Counter-rotating**



Two identical screws that rotate in parallel in the same direction.

• The material goes alternatively from one screw to the other and thus mixture is more homogeneous than in counter-rotating screw extruders.

• Screw configuration, barrels and feeders are designed depending on the kind of filler, fibre, polymer, percentages of each component.

• <u>These type of screws are usually used in long extruders, with a high L/D (length/</u> <u>diameter) ratio</u>. Due to the low pressure with which these extruders work, the addition of liquid additives by peristaltic pumps is an easy process.

• <u>The flow dependence on material viscosity is higher than in counter-rotating screw</u> but lower than single screw extruders.



• In this kind of extruders <u>the flow generated has a *C shape*</u>, which has the properties of a positive pumping, decreasing the influence of material viscosity in its conveying and <u>generating a very effective pumping</u>.

Some drawbacks of this process are:

- Screws are pushed against the barrel, which makes necessary to a void high screw speeds.
- <u>Inefficient mixing</u>. The faster the material is conveyed, the less <u>efficient the mixing is</u>.

## Application to compounding operations

- The main application of twin-screw extrusion is compounding, that is, the mixing of a solid filler with a molten polymer phase, to obtain a material with improved properties
- <u>Distributive mixing</u>, which consists of homogenizing the spatial distribution of the filler into the matrix and it is related to the kinematics of the flow. The more or less complex trajectories of particles allow them to be distributed homogeneously in the matrix
- <u>Dispersive mixing</u>, which consists of reducing the size of the filler, is instead connected to the state of the stresses that, when applied to the filler, allow them to break the particles and reduce their size
- In an industrial compounding operation, both types of mixing are generally involved. It is generally desired to first reduce the size of agglomerates or aggregates to the lowest possible level, which will give the best ultimate properties, and then obtain a spatial homogeneity of these fillers.
- Distribution is attained through the re-orientation of the flow by screw elements, while dispersion depends on the local stresses.
- The above considerations explain why a co-rotating and intermeshing twin-screw extruder is the most effective to ensure the correct mixing of two molten polymers in order to produce a blend, due to the high concentrations of the minor component. In fact, this configuration is able to provide the highest possible elongational deformation and to multiply the shear stresses undergone by the polymers thanks to the combination of two parallel screws.









- (a) poorly dispersed, poorly distributed;
- (b) poorly dispersed, well distributed;
- (c) well dispersed, poorly distributed;
- (d) well dispersed, well distributed

## Twin-screw extrusion complete equipment

• To better understand the extrusion compounding process, we must know the main components of a compounding extruder. Here there is a global scheme of a common compounding extruder and its main components.



#### What kind of products can we extrude?

Different **kinds of compounds** can be made depending on which kind of material we add to the polymeric matrix. Blends or compounds that we can obtain by extrusion compounding are:

Polymer / polymer blends

Polymer / additive compounds

Polymer / filler compounds

Polymer / reinforcement compounds



#### POLYMER / POLYMER BLENDS

This kind of blends are **PHYSICAL BLENDS**. Almost all of these blends are **incompatible** and hence, **domains** of the polymer which is the minor component, are created in the other polymer, which is the major component.

For immiscible polymers, the mixing process results in a specific morphology, controls the properties. Depending on the composition and the interfacial and rheological properties of the two phases, the mixing of two polymers A and B will give several morphologies.



#### POLYMERIC COMPATIBILIZING AGENTS

- As these kind of blends are **incompatible blends**, an <u>interfacial tension</u> between the surface of both polymers is created. With the purpose of decreasing this tension and increase adhesion between polymers, **compatibilizing agents** are added to polymer blends. Thanks to compatibilizing agents, the possibility of improvement of blend properties increases. Besides, it usually yields **properties that are generally not attainable in either single pure** component.
- Compatibilizing the system will make a more stable and better blended phase morphology by creating interactions between the two previously immiscible polymers.



#### POLYMER / ADDITIVE COMPOUNDS

These kind of blends are **necessary**, not only to **improve the processability** of polymers in many of their manufacturing processes, but also **to achieve a good performance** in almost all their final applications.

Additives that can be added are:



• nucleating agents

• plasticizers

- flame retardants
  - antioxidants
- chain extenders



Some of these fillers are: calcium carbonate, precipitated carbonate, talc, mica, kaolin, synthetic silica, barium sulphate, alumina, antimony trioxide, etc.

## Going in depth...Compounding extruders

• The compounding is an extrusion-pelletization process that allows the mixing of a polymer by melting (thermoplastic resin) with one or more solid or liquid additives in order to obtain a physical blending of the ingredients..





- 1. Dosatore
- 2. Dosatore opzionale
- 3. Forzatore per la carica laterale (Side stuffer)
- 4. Forzatore per la carica laterale (Side stuffer)
- 5. Estrusore bivite corotante
- 6. Degasaggio
- 7. Taglio in testa



Schema linea di estrusione per compounding (Comac S.r.l.)

## Going in depth...Masterbatch Extruders

- A particular type of pellet is the masterbatch, which consists of a pellet with a high concentration of additives (such as colorants), compared to the desired concentration of these additives in the final product.
- The masterbatch is blended with virgin polymer pellets during the extrusion process to facilitate the addition of the additives. Without the use of masterbatch, additives would have to be added in the form of powders or liquids, which presents obvious dosing difficulties.
- Indeed, it is much easier to accurately weigh a few grams of masterbatch than to achieve the same weighing precision with a few micrograms of powdered additives.



## Going in depth...Recycling Extruders

- Recycling extruders have been primarily developed for the recycling and recovery of "waste" plastic. Having already undergone an extrusion process, the recovered material has still undergone some degradation and cannot be extruded in its pure state. However, depending on the type of material, it can be reintroduced in variable quantities, depending on the product itself and what is desired as the final product.
- For example, some PET water bottles are produced with a percentage of recycled PET, although the virgin PET must still account for more than 65%. Recycled material affects the transparency of the virgin material (it becomes yellowish), so it is always extruded with a high percentage of colorant. In fact, colored plastic bottles for mineral water are obtained from extruded virgin PET + recycled PET + coloring masterbatch (usually green or blue).





## **Going in depth...Hot Melt Extruders**

- Hot melts are thermoplastic solid materials which are inherently solid below 80 °C and they become low-viscosity fluids at higher temperatures. Typical production is the melt coating.
- In pharmaceutical production, HME (Hot Melt Extrusion) is used to disperse Active Pharmaceutical Ingredients (APIs) into a molecular-level matrix, thus forming solid solutions. This enables drug release systems for poorly soluble drugs or specialized drug forms such as films for transdermal patches.



Schema di estrusione per Hot Melt (Thermo Fisher Scientific)

## **Going in depth: Bioplastics Extruders**

- The extrusion process of bioplastic requires specific knowledge of this particular material, with fluidity often varying from one production batch to another and depending on the type of compound.
- Furthermore, it requires the study and construction of specific extrusion screws for this material, as well as the use of dies with appropriate compression to achieve a homogeneous melt capable of ensuring constant and uninterrupted productivity.
- Here an example for the extrusion of the starch:



Schema linea di estrusione per bioplastica (Coperion GmbH)

- 1. Dosatore/premiscelatore amidi
- 2. Dosatore plastificante liquido
- 3. Alimentazione polimeri in pellet
- 4. Estrusore bivite corotante ad alimentazione laterale
- 5. Degasaggio
- 6. Degasaggio sottovuoto
- 7. Testa
- 8. Vasca di raffreddamento
- 9. Lama d'aria di asciugatura
- 10. Pellettizzatore

## Lab Extruders

- The laboratory twin-screw extruder represents the ideal solution for all those users who want to test materials or processes without employing a standard plant with high energy and material consumption.
- These users include research centers, universities, laboratories, training centers, as well as manufacturing companies that want to test extrusion processes on a small scale to evaluate the feasibility and quality of the extrudate before proceeding with actual production.







Estrusore bivite da laboratorio (Labtech GmbH)



## How are made the screws

- Most of the machines are **modular**, in that they consist of small elements of fixed length, which can be arranged on a splined shaft to obtain a certain screw configuration.
- The design of a screw configuration for a defined application is often made by a trial-and-error procedure based on experience and rational guidelines.



- Conveying elements, either direct (right-handed) or reverse (left-handed)
- Kneading disks, with one, two, or three lobes with the same cross section as the corresponding screw elements
- Mixing disks such as gear wheels, designed to separate and recombine the material flows.

### PROCESSING UNIT: SCREW ELEMENTS

It is possible to list the characterization and the mode of operation of extruder screw elements.

• It is a geometrical quantity and influences the chamber volume and residence time.

• It describes the available volume in the screw flight.

• It becomes possible by the "Erdmenger profile" of the screws and is ideal for conveying elements.

• The **residence time** of the material **in the screw** is determined by the pitch (the higher the pitch is, the shorter the residence time will be): However it is important to observe that the **residence time in the extruder** is determined by the screw speed and the length of the processing unit!

• It describes the element's ability to push the material forward. The more flights there are and the smaller the pitch is, the stronger the conveying effect will be.



shaft The screw F, F<sub>M</sub> hub allows transmission of high torques.  $\alpha = 30^{\circ}$  $F_t = \cos \alpha \cdot F_M$ shaft  $F_r = \sin \alpha \cdot F_M$ 6 M



**Kneading and mixing elements** allow the dispersion and distribution of loads and additives inside the polymer matrix. They are placed in the melting and mixing zones.

The **pitch** of the screw is the distance between two consecutive flights. The screw flight pitch is directly related to the screw helix angle, which is the angle between the screw flight and the plane perpendicular to the screw axis.



**High performance elements** can lower the energy input into the product with very high distributive and dispersive mixing action.



**Conveying elements** their mission is to make the material advance through the screw, for this reason, they are placed in the feeding, degasification and metering zones.

Pitch

Chamber

volume

Self-cleaning

Conveying

effect
## More on...Screws elements



#### **Conveying elements Example of Identification: C-D-2-30-60**



#### Mixing elements Example of Identification: M-D-2-30-60



#### Kneading blocks (or disks) Example of Identification: K-D-2-5-60°-60



## More on...Screws elements

- <u>The pitch varies between different modules</u>, since <u>a larger one is</u> <u>characterized by lower residence time and transport efficiency</u>
- *Inverse elements* are special modules with a reverse helical thread that can be employed if it is necessary to increase the residence time in a particular section of the screw

• <u>They are used to re-orientate the flow</u>, in order to get distributive mixing or <u>to improve the intensive mixing provided by kneading</u> <u>elements</u>. They are substantially screw elements with slits cut on the threaded edges following a helical path

• They are constituted by a sequence of bilobal or trilobal discs put aside with a certain staggering angle (commonly 30°, 60° or 90°), so they have an helicodal shape although they do not show any thread.

These modules have the task of providing melting, thanks to the high shear rate, but also extensive and intensive mixing because the clearance between two lobes is very small

 $https://www.youtube.com/watch?v=qM1dfuy-qNc\&ab\_channel=GammadataInstrumentAB$ 

## **Processing unit: Extruder zones**

The representation of conveying elements and kneading elements in a screw configuration draw is easy. Conveying elements are represented as a diagonal line and kneading elements are represented like a stair.

In a co-rotating twin screw extruder it is possible to individuate six different zones:

1.	Feeding zone	4.	Mixing zone
2.	Melting zone	5.	Degasification section
3.	Secondary feeding zone	6.	Metering zone

It is the last section, its C mission to provide q pressure to the melted material to make it go 60 through the die. Usually the last element posses a lower pitch in order to provide a higher pressure to make the material go through the die.



### **Extruder parts: Barrel elements**





- <u>As the screws, the barrel sections are modular and can be</u> <u>arranged in any configuration necessary to accomplish a</u> <u>particular extrusion objective.</u>
- Barrel sections normally have either **rectangular or circular** outside dimensions depending on the manufacturer. Each barrel section contains a thermocouple to control the heating and cooling input. Barrel sections have flanges on each end for alignment and connection to the next barrel
- The vent barrel has an opening on top that may be either circular or rectangular and is used to vent volatiles from the barrel or to feed different components of the formulation.
- The first barrel section in the extruder is opened on top for feeding all or part of the formulation into the extruder. It is cooled and normally has no heaters on any of the sides.





## **Peculiar Barrels: Half opening**

 In small-sized extruders such as those for pilot lines or laboratory use, it is preferred to use extrusion barrels in two halves, which can be opened by means of lateral hinges. This simplifies the necessary mechanical operations and reduces machine assembly and cleaning times.



## **Extruder Parts: Feeders**

#### **Primary Feeding**

- The main forced feeding is a motorized screw conveyor that takes polymer pellets (or powder) from the loading hopper and pushes them, forcing them to enter the feed throat.
- The correct method of polymer feeding for a twin-screw extruder depends on the design of the twin-screw extruder, the fed material, and the feeding position. In counter-rotating and co-rotating parallel twin-screw extruders, polymer and feeding additives are fed "hungry-mouthed" into the feeding section of the extruder.



Forzatore per l'alimentazione di un estrusore bivite

### **Extruder Parts: Feeders**

- Feeding means adding or discharging a certain quantity of material to or measuring process (in case of continuous mixing) throughout a defined time frame.
- The feeder typology must be chosen according to the type of material (pellets, powders or fibers) in fact, they can influence the dosing ability and accuracy



components

Vibrating tray feeder

#### PROBLEMS TO AVOID

- **Product Bridging (or "arching") and rat-holing** are both issues that result in a *no-flow condition*. Bridging, is a case where material that is being discharged or fed forms a bridge or arch over the feed auger or discharge point in a silo cone/hopper. Rat-holing is a condition where the material forms a hole or narrow channel above the feed auger or outlet in a hopper while the remaining material is stationary against the hopper wall.
- Both of these conditions result in the product not flowing as desired.





## Screw configuration: degasification section

- The extrusion process of a polymer can generate water vapor due to the moisture present in the pellets. Some polymers, during the granule melting phase, also release gases (e.g., chlorine in the case of PVC) or monomers that are highly toxic.
- If these vapors and gases are left inside the molten polymer, they would advance to the exit on the extrusion head, causing deformations and defects in the final product, making it unacceptable in terms of quality and functionality. Therefore, they must be removed, significantly improving the physical and structural properties of the product.
- The degassing device is realized by installing one or more venting ports on the extrusion barrel, which are usually connected to a vacuum pump that facilitates extraction and prevents dispersal of toxic gases into the atmosphere.
- The vacuum pump sends the extracted gases from the extrusion barrel to a condensation tank. At the base of the tank, condensate from the hot gases is collected, while at the top of the tank, a duct sends the gases to the filtration and purification system, usually installed by the customer according to local regulations.
- The solution of installing degassing ports (which can be up to three or four on the same extrusion barrel) is preferred by processors who do not want to dry the granules before feeding them into the hopper and by those who use polymers that produce gases and monomers during the polymer melting process.



### **Processing units: dies**

The role of a die is to form the melt into a desired linear product: fibres, films, sheets, profiles, rods, etc. The die is a channel, whose profile changes from that of the extruder bore to an orifice, which produces the required form.

For extrusion compounding, **die head has usually a circular shape**, in order to obtain "spaghetti" filaments, which are then cut into pellets. However, the number of filaments and thus, the total production that can be achieved with an extrusion compounding extruder changes depending on the number of holes of the die head.





- ✓ A Typical Die Design for extruding a solid strand is reported in figure.
- ✓ In the figure,  $D_D$  is the diameter of die orifice,  $D_B$  is the diameter of bore of extruder barrel,  $\alpha$  is the lead-in angle, and P is the die land.
- ✓ Because of the screen pack and breaker plate assembly, the pressure in the extruder ( $P_E$ ) is reduced by the pressure loss across the assembly ( $P_L$ ).
- ✓ Since the die outlet is at atmospheric pressure, the working pressure is the die pressure ( $P_D$ ) given by the difference:  $P_D = P_E P_L$ .

#### 4 6

### Extrusion process exiting from the die

 After the mixing and melting the material exits the die by pressure in tubular shape (melted filaments). Such melted filaments go through a water bath where they are cooled (in this case) and, once cool, they exit the bath and are dried and stuffed into a pelletizer where they are cut into pellets, which can be used in other manufacturing processes.



• During the pelletizing, the polymer properties are "frozen"; in addition, the shape size and weight are fixed. Pelletizing problems are mainly related to the speed selection. Pelletizer speed must be selected depending on the extrusion speed. If pelletizing speed is not correctly selected plastic filaments can be broken, if speed is too high, or material is accumulated in the melted filament, if speed is too low.



### **COMAC extruder at Pisa University**



- Co-rotating twin screw extruder with D=25mm and L/D ratio= 44.
- Four feeders, degassing pump, circular nozzle, water bath and pelletizer





### Going more in depth: Necessity to optimize the process

- <u>Very often the identification of the best composition is carried out with an empirical process with a twin-screw extruder and the final characteristics are then evaluated.</u>
- <u>This definitely lengthens the development time also because, in addition to the composition able to guarantee the best performance, it is necessary to identify the screw profile that allows the best final microstructure to be obtained.</u>





It can be useful a tool to optimize the processing parameters!

## Parameters to optimize for the extrusion



- Process control parameters for a twin-screw extruder include screw speed (revolutions per minute), feed rate, process temperatures, and vacuum level (to aid devolatilization). Additionally, melt pressure, melt temperature, motor amperage, and various in-line sensors also monitor the process to ensure consistent and quality product.
- Is it possible to optimize these parameters saving materials, save energy, avoiding waste and earn money?

## **Temperature profile**

- Only some temperature profiles can give rise to an acceptable product while simultaneously providing a process under optimal conditions. When one knows where the polymer melts or softens, the required mixing after melting and any downstream feeding and venting openings, it is possible to select temperature set-points based on processing requirements for formulating a particular polymer. Possible temperature profiles include:
- Progressive temperature increase, with set-points continuously rising from the feed throat to the extrusion head.
- An inverse or decreasing temperature profile, with set-points decreasing from the first zone of the heated cylinder to the extrusion head.
- Constant temperature profile, where all cylinder zones are set precisely at the same temperature set-point.
- A Gaussian curve profile, where the temperature is lower in the first zone of the heated cylinder, then gradually increases towards the central zones of the extruder before decreasing progressively towards the extrusion head.

## **Residence time concept**

• The residence time, or polymer residence time, is the duration during which each polymer granule or particle remains inside the extruder during the process; the time is considered from entry into the feed throat until the exit point, conventionally defined as the terminal flange of the extruder or at the exit of the die.

Each screw profile is associated with a polymer residence time that depends on several factors, such as:

- □ Number of screw revolutions.
- □ Number of conveying screw sections.
- □ Number of mixing screw sections.
- □ Number of rear conveying screw sections.
- □ Number of other screw sections.
- □ Material production rate in kg/hour.

- The residence time in the extruder is an extremely important factor as it relates the temperature curve of the polymer and its degradation; a polymer can be heated for a long time at low temperature or for a short time at high temperature without degrading.
- If, on the other hand, the dwell time at high temperature is too long, this will certainly cause the polymer to degrade. Longer dwell times produce such degradation of the polymer that it returns to a solid state at high temperatures and consumes the thermal stabilisers.



## **General Rules**



- N = Screw Speed, rpm
- Q = Throughput Rate, kg/hr
- SEC = specific energy consumption, kWh/kg

### Ludovic Software



Ludovic<sup>®</sup> is today the virtual extrusion lab designed for optimizing the corotating twin screw extrusion process. Ludovic<sup>®</sup> provides the material sensitivity for rationalizing & cutting down experiments.



#### Virtualizing the extrusion process

Modeling the extrusion process for

understanding the material behaviour during the process
getting reliable data about the material/process history

## What is it ?



## A simulation software



## Dedicated to corotating twin screw



## For all the corotating twin screw exturders

Of any diameters or

brands

To pilot From Micro or Lab scale And industrial line

10 100 100

## The modeling : principles



Reproducing a physical phenomenon by a mathematical model





## **LUDOVIC Requirements & Outputs**

• Ludovic requires the definition of various operating parameters (detailed information are given later) and after the calculation <u>it reports</u> data about energy consumption, residence time distribution and many other quantities, like temperature, pressure, viscosity and so on, as *a function of time or of the axial distance from the die.* 

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- Data can be exported as a .xls file which can be elaborated and compared with the results of other simulations.
- Using the settings of a specific simulation, it is also possible to define a **Design of Experiment (DoE)**, which basically consists in the automatized repetition of that simulation by progressively varying one or two independent parameters defined by the user. <u>This procedure allows to find the conditions needed to reach certain desired values without the necessity to proceed by attempts, that is a more time-consuming approach which does not ensure to achieve the goal.</u>



## The Ludovic<sup>®</sup> modeling : principles



### For Ludovic<sup>®</sup>, input data are



## The interest of modeling - different focuses

- 1 Simulation
  - For checking
  - 1 set of operating conditions
- Comparison of simulations
  - For optimizing
  - Few sets of data
- 1 DoE / QbD
  - For anticipating
  - Hundred of Simulations
  - Covering a functioning domain
  - Example : new formulation



## The simulation window



### **Geometry components - Organization**

The geometry description of the extruder is organized into 5 topics

- Extruder dimension : diameter, Ro/Ri ratio,...
- Barrel definition
- Profile of the extruder : screw elements
- Die description

• Feeding area





## Extruder dimension





### **Dispersive and Distributive Mixing**





## Possibility to create non existing screw element



## Screw element export

### Export to the library



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## Die definition as a Lego

Die = Thermo-mechanical boundary condition for the material



**i** Die Elements X 20.00 L:Length (mm) Cl:Centerline (mm) 18.80 Di:Diameter (mm) 24.00 Converging Slit 8/0 De:Diameter (mm) 8.00 Filter Imposed p essure Modify

A pressure can be imposed

## Products

### Definition

Different components are available in the [*Products*] tab. Each of them holds a specific function.

#### Products

Means the main material matrix.

It is either a single material or a blend.



### Recipe

Means a between

- combination
- two products or more
- product(s) with additives



### Additives

Means solid particles (do not melt). As additive, their combination with a product or a recipe is computed with a relative viscosity model.



## **Technical Bastions**

There are many types of formulations that can be obtained with the twin-screw extruder, but the *"technical bastions"* that must be considered in order to obtain a good product are:





### In Ludovic<sup>®</sup>, a material is defined by its



The approach of Ludovic  $\psi$  consists in considering a local description of the flow field of the molten material in each element of the screw profile, from hopper to die.

Along the screw profile, the flow field is resolved by elementary models dedicated to each type of element. In each elementary model, the physical results are calculated <u>depending on the material parameters</u> (thermophysical and mechanical properties) and process parameters (flow rate, rotation speed and thermal regulation parameters).

The heat balance is also considered in each element. This heat balance includes the dissipated power and the heat transfer between the molten material and the barrel, screw or die.

→ Thermal and mechanical data are thus used for providing a description of the material evolution along the screw profile



## The thermal characteristics

The thermal characteristics requested in Ludovic<sup>®</sup> are

Heat Capacity (Cp – in J/kg/°C)

Means the amount of heat needed (Q) to raise the temperature of a unit of mass (m) of the material by one degree of temperature :

# $Cp = \frac{Q}{m \,\Delta t} = \frac{q_m}{\Delta t}$

### Density (ρ – in kg/m<sup>3</sup>)

The volumetric mass density ( $\rho$ ) of a substance is its mass (*m*) per its volume (*V*). It is defined by :

#### Thermal conductivity (λ in W/m.K)

- The property of a substance to conduct heat. It is primarly evaluated in terms of Fourier's law or heat conduction.
- Melting temperature (°C) and melting enthalpy (kJ/kg)
  - Melting Temperature: temperature at which a substance changes from solid to liquid phase
  - Melting enthalpy : energy provided by a substance when changing from solid to liquid phase

 $\rho = \frac{m}{V}$ 

### The thermal characteristics

In Ludovic<sup>®</sup>, the thermal characteristics are split into :

- Solid state
- Liquid state

Thermal Characteris	tics Viscosity		
Exa	ample of a classic P	ΡE	
Solid Phas	e		
	Heat Capacity (J/kg/°C)	2300	
	Density (kg/m3)	920	
	Thermal Conductivity (W/m.K)	0.33	da
Liquid Pha	se		
	Heat Capacity (J/Kg/°C)	2730	To
	Density (Kg/m3)	920	Fc
	Thermal Conductivity (W/m.K)	0.33	b
	Helling Terrorekurg (10)	110	da
	Metting Temperature (°C)	119	
	Melting Enthalpy (KJ/Kg)	220	
(*) Optional for the Fi	low Computation		

o be determined at ambiant temperature

If no data available for the solid phase, liquid phase input data can be used.

#### To be determined at "process" temperature

For following the material during the process, data have to be representative of the material evolution. The closer the data are, the better the results.



## • The thermal characteristics

Thermal transitions of a substance can be obtained by using a DSC

#### Some definitions

- Differential Scanning Calorimetry
  - Is a thermoanalytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference is measured as a function of temperature. Both the sample and reference are maintained at nearly the same temperature throughout the experiment. Generally, the temperature program for a DSC analysis is designed so the sample holder temperature increases linearly as a function of time. The reference sample should have a well-defined heat capacity over the range of temperatures to be scanned



The thermal characteristics can be obtained by using a DSC

#### Melting temperature

1

Melting temperature is availble with this kind of curve



P12
# The obtained data by a DSC The thermal characteristics can be obtained by using a DSC

- Heat Capacity (Cp in J/kg/°C) ٠
  - The obtained heat capacity looks like the hereafter curve. 3 materials are compared on this figure
- The Input data for Ludovic
  - As a reminder, we select data included in the adapted process functioning domain
  - Considering a material processed at 120°C; chosen data will be the following ones :



Cp for the liquid phase : selected at the "process" temperature :

- 1650 J/kg/°C
- 2300 J/kg/°C
- No available data in this case



### The rheological characteristics

In Ludovic<sup>®</sup>, mechanical characteristics are defined from different ways



Can be measured by a **capillary rheometer** 

\*As analytical laws can be found in literature (or on specific database), we will focus in this Quick Guide on the SOP method.

# **Experimentally...Rheometry!**

It is important to evaluate rheological behavior of polymer melts: *rheometry* is the field of science that studies the instrumentations and facilities to evaluate the measurement of rheological data.



Shear flows vs Extensional flows - Pressure flows vs drag flows - Steady-state vs dynamic state –Large deformations vs Small deformations

# The PROCESS tab

The PROCESS tab allows to define the operating conditions



Operating conditions

- 1. Setting a screw speed
- 2. Associated a product/recipe to the appropriate feeding zone
- 3. Setting a throughput for the feeding zone
- 4. Setting an input temperature for the product/recipe
- 5. Setting a temperature of regulation for the barrel
- 6. Setting a temperature of regulation for the die (optionnal)

# HOW TO ANALYZE THE RESULTS?

### Global values

Energy consumption and distribution

Torque and power

Global residence time, ...

Residence time distribution

Evolution of the thermo-mechanical values along the screw

(Temperature, Pressure, Pressure drops, ecc.)

Evolution of Temperature, Pressure and Shear rate with residence time

[Global Results tab]

[RTD Results tab]

[Results f(x) tab]

[Results f(t) tab]

# **Energy distribution**

Specific mechanical energy (SME) = Viscous dissipation energy (screw + die) +Solid transport energy + Melting energy

Product energy = SME + Conduction energy

Extruder energy = SME + *absolute\_value*(Conduction energy)

Mean residence time	=	49.4	s
Dissipated Energy (viscous dissipation - screw)	=	148	kWh/t
Specific energy (solid transport - screw)	=	53	kWh/t
Melting Energy (screw)	=	61	kWh/t
Dissipated Energy (viscous dissipation - die)	=	2	kWh/t
Total Conduction Energy (Screw+Die)	=	-83	kWh/t
Total product Energy	=	181	kWh/t
Total extruder Energy (abs. value of conduction)	=	347	kWh/t
Power engine	=	3	kW
Torque / Shaft	=	101	N.m
SME	=	264	kWh/t
Conduction power / Barrel			
Barrel n° 1	=	0	kW
Barrel n° 2	=	0	kW
Barrel n° 3	=	0.0606	kW
Barrel n° 4	=	-0.2078	kW
Barrel n° 5	=	-0.3392	kW
Barrel n° 6	=	-0.2489	kW
Barrel n° 7	=	-0.0935	kW
Barrel n° 8	=	-0.1708	kW
% Dissipated Energy (viscous dissipation - screw)	=	42.74	&
% Specific Energy (solid transport - screw)	=	15.12	8
<pre>% Melting Energy (screw)</pre>	=	17.6	&
% Dissipated Energy (viscous dissipation - die)	=	0.56	&
<pre>% Total Conduction Energy (Screw+Die)</pre>	=	23.99	ঞ



# Residence time distribution (RTD)



# The RESULTS f(x) tab



The RESULTS f(x) tab allows to display the material thermomechanical history, along the screw profile

The results:

- 1. Temperature evolution in red
- 2. Pressure evolution in blue
- 3. For selecting/unselecting other results

# The RESULTS f(t) tab



The RESULTS f(t) tab allows to display the material thermomechanical history, as a function of the time

#### The results:

- 1. Selection of the temperature
- 2. Temperature evolution as a function of the time (does not refer to the screw profile at the bottom)
- 3. Setting a temperature threshold

# **Simulations Results Comparison**



# A case study

How to decrease the length breakage of carbon fibers during the production of short fiber composites by twinscrew extrusion?







The first step regarded the design of a simulation, using the software Ludovic®, that accurately replicates the original process, as previously performed.

Subsequently, three variations of this simulation were designed and are proposed in this presentation:

ξΞ

- Matrix material: 80% PA66 + 20% CF → Version 1
- Screw configuration
  - Different kneading zones arrangement → Version 2
  - Different kneading zones arrangement + different position of the feeding zone → <u>Version 3</u>



Lastly, DoE analysis was performed on the original setup of the process.

### Materials Matrix

### **PA6**

Zytel® 7335F NC010

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	$_{2})_{5} - C - \frac{1}{n}$

Melting temperature [T <sub>m</sub> ]=°C	221
Degradation temperature [T <sub>max</sub> ]=°C	>300
Glass transition temperature [T <sub>g</sub> ]=°C	60
Theoric degree of crystallinity DoG, %	35
Tensile modulus [E]=GPa	3.6
Recyclability Y/N	Yes



### **Fibers**

#### Zoltek® PX35CA (Chopped Fiber)

Carbon chopped fibers are commonly compounded with general engineering thermoplastics (such as PC and Nylon) and high-temperature thermoplastic resins (such as PEEK and PEI). The resulting composite offers high strength-to-weight and stiffness-to-weight ratios.

- high bulk density value ightarrow cleaner and more consistent flow rates
- easy distribution during compounding ightarrow improved process and product performance





# Simulation Results f(x) results

Temperature, °CPressure, Bar







Average fiber length, µm

# Varying Process Parameters: Conclusions



Being the main aim of this project the maximization of the average length of the CF in the material exiting the process, <u>Version 3</u> (different feeding zone and screw geometry) appears to be the best choice since the fiber mean length is 4252,53  $\mu$ m (vs. the initial length of 6000  $\mu$ m) and the melt temperature exceeds the degradation temperature of the matrix material (300°C) for less then 4 seconds.

# Varying Process Parameters: Conclusions



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# DoE

The DoE analysis was performed on the original process (Version 0) to achieve optimization of the process parameters. More in particular,

- rpm (primary parameter) → [150,350] tr/min → 11 computations
- flow rate, feeding zone #1 (primary parameter)  $\rightarrow$  [8, 24] kg/h  $\rightarrow$  11 computations
  - flow rate, feeding zone #2 (fiber input) (secondary parameter)  $\rightarrow$  [10, 30] kg/h
  - flow rate, feeding zone #3 (degassing) (secondary parameter) → [9.9, 29.7] kg/h



# **DoE: Conclusions**

 ${\rm Goal} \rightarrow {\rm maximizing}$  the average fiber length

Values taken into account  $\rightarrow$  rpm + flow rate at feeding zone #1 + torque + max temperature + SME + RTD



Avg fiber lenght	rpm	Flow rate	Torque	Max temperature	SME	RTD
μm	tr/min	kg/h	N·s	°C	kWh/ton	S
4252,530	150	19,2	187,5	299,61	273,4	36,66
4222,175	150	20,8	197,4	297,04	266,2	35,43
3859,520	350	24,0	141,0	397,61	380,5	20,96
2609,960	310	8,0	81,26	412,68	577,5	45,78



More than a substance, plastic is the actual idea of infinite transformation

**Roland Barthes** 

Life in plastic...it's fantastic!

Aqua





