



# ASSESSMENT OF BLENDS OF SBS COPOLYMER WITH POLYETHYLENE FOR FILM APPLICATIONS



**PFC - 112121**

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## **1. INTRODUCTION**

**Title:** Assessment of blends of SBS copolymer with polyethylene for film applications.

**ID number:** 112121

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**Location:** Develop in Dow Chemical Company (Tarragona).

**Date:** 2011/2012

**Summary:** Aquest projecte pretén estudiar mesclades de polietilè amb *Styrene-Butadiene-Styrene (SBS)*. La idea és recopilar la literatura necessària per tal de desenvolupar noves mesclades i estudiar-ne les propietats a fi d'observar-les i crear un producte nou i diferent per portar al mercat. Més concretament, el que es persegueix és millorar l'equilibri entre propietats mecàniques i òptiques dels films.

Per tal de produir les mesclades s'utilitzen línies d'extrusió i, en aquest cas, presenten tot una sèrie de limitacions degut a la refrigeració i aquesta serà la part que s'estudiarà en l'apartat de disseny. L'objectiu que és millorar la refrigeració de la massa polimèrica a fi d'incrementar encara més els kg/h i poder extruir les mostres en unes condicions més similars a les industrials de gran escala. Es presenten tot una sèrie de mesures que es comparen entre sí per determinar quina seria la millor opció. A més, també inclou el disseny d'una d'aquestes opcions. Es tracta d'un sistema de refrigeració interna que s'anomena *Internal Bubble Cooling*.

Finalment s'avalua econòmicament l'efecte de la implementació d'aquestes millores en la línia actual a fi de retallar costos al mateix temps.

“Thank you very much for the help and feedback provided by Jesús and Toni”

“Thanks to Marta for the assistance with English”

“And thank you so much to Anna for the support”

## **2. PRELIMINAR STAGE**

### **2.1. Project description**

In this project we want to assess the blends of the Styrene-Butadiene-Styrene (SBS) copolymer with polyethylene (PE) for film applications. We can find many references in the market and scientific literature, therefore, the final objective is to review the existing knowledge and try to find something different, new and useful for the polyethylene markets. The idea is to avoid repeating experiments to see the improvements or worsens; the aim is to improve the mechanical-optical properties balance of the PE films.

Dow works this way because nowadays, this balance is one of the most important points in the plastic industry. Many companies work to improve the optical properties of their products without worsening the mechanicals and the balance can still be improved.

It is not usual to commercialize a pure PE for an application because, when we blend it with another well selected material, the properties improve and it depends on the application it is blended with one material or another. Here we are facing an essential point; whoever develops a product which presents the best property balance will be able to gain customers. Dow's research line is focused on studying how specific properties of the films can be improved in order to offer their customers the best products for their application with the best service and know-how so as to pursue the aim of becoming the best chemical company with the best products and services. However, Dow is defined by more than their products and services. The history and the company strategy, their organizational leadership, their people, all of these criteria are the main reasons which define the company's culture.

### **2.2. Project scope**

This project is constituted by two parts. One is based on research and development and the other constitutes the designing part of the project. In reference to the first part, we can identify two clear objectives:

- Search information and scientific articles about polyethylene-SBS blends and some compatibilizers.
- Propose new blends with polyethylene after selecting the appropriate product, in theory SBS.

Both of them constitute the research part of the project which is very important and is the cornerstone of the project around which everything takes meaning. But there is a larger part

which consists of selecting the products and the ideal combinations in order to find new blends which present better properties than other blends studied up to now. But to achieve the above mentioned goals, first, we need to clearly identify past experiences and new potential opportunities.

The first step is to know what kinds of blends have been done, and then, the idea is to define a group of samples to extrude. In contrast, the construction part is focused on the design of an extruder. It implies the following items:

- Study the area and its climate, natural resources, energy prices and legislation.
- Design the parts of one extruder and the corresponding process diagrams.
- Elaborate the basic design and control systems with the appropriate instruments.
- Describe a security plan so as to protect the extruder, facilities and employers.
- Carry out an environmental study of the facilities where the process takes place.
- Establish a maintenance plan to protect and safeguard the installations.
- Create an operation manual giving details about the equipment and its operation process.
- Develop an economic study to know the process profitability.

To extrude them we will use blown film extrusion equipment which includes one single extruder and it can manufacture a 22.5 cm wide film. This equipment is design to work at 4-5 kg/h. The main problem which presents is the thickness distribution because in comparison with the blown film coextrusion machine is worse. But, in contrast, the machine's availability is the main reason to do the monolayer studies there. It's faster and the deviation is within 10% of error, therefore acceptable.

Referring to the following study, which consists of a multilayer film, the coextrusion machine includes 5 extruders with different feed-blocks which can make up to 7 layers. The equipment was designed to work at 30 kg/h, even though the cooling system does not have enough capacity and it can only work with an output of 15 kg/h at most. We know that this is the weak point which we need to study if we want to work with a higher output; therefore, we can think of alternatives or complementary cooling systems. This would be the objective of the following part:

- Suggest different alternative cooling systems and design them in order to extend the cooling capacity of the current system so as to work at 30 kg/h.

One possibility would be implementing an extra cooling system to cool the internal part of the bubble because it only has an external cooling system now. This additional system is called Internal Bubble Cooling (IBC) and it could be the solution so as to extrude with a higher output. There are different kinds of IBC and we will have to choose the best option or suggest different and compare them. We will have to take into account the old and new systems and compare the cooling capacity of each one. Apart from studying the temperature gradient we could try to extrude with different products and mixtures. The final idea would be to imagine working in the worst conditions in order to extrude the blends more easily. However, the main problem is the cost. The extrusion machine has a small die diameter and in this case it is nearly impossible to implement it at an acceptable cost. There are other alternatives such as pre-cooled air from a separate heat exchanger and/or a more sophisticated refrigerated ring. The alternative of using a heat exchange, which was implemented 3 years ago, has achieved an output of 12-15kg/h. The other can be a good option, but first it has to be found on the current market and its convenience must be estimated.

In case of IBC, we should design the parts of this additional system and adjust all the parameters with the worst conditions to achieve the highest output ensuring a good quality films. Now the weak point of the equipment is the cooling system, therefore, the objective is to implement a new system and optimize its functioning. All of these alternatives are economically evaluated in order to see if they are profitable or not.

In addition, we will include an equipment operation manual explaining the main steps to follow if you want to start-up or stop the procedure. And we will explain the operation and the parameters we can manage. Referring to the new implemented system, we will explain the improvement we can achieve with it and the influence of them in the final product.

Moreover, we will link it with the safety measures and all the convenient point relating to protect the equipments, installations and the personal who work there. We will pay especially attention in this part because is one of the essential points together with the maintenance and economy. These will also be included in the project because first we need to know if the idea, the change is profitable and the extruders and the other part require a maintenance plan to protect and safeguard the installations.

Apart from all these parts, before manufacturing the samples it is necessary to calculate the corresponding amounts to prepare. Once we have selected and prepared the blends we

want to extrude and the samples are manufactured, we will have to test them in the laboratory and will have the values to determine if the extruded film has the mechanical-optical property balance which we are looking for. But, to extrude the film will not be easy because we have never used this product (SBS) in this machine and have no idea about the operation conditions that must be used in order to manufacture a stable bubble and a good quality blown coextrusion film. Here, the cooling system improvement can develop a crucial role because we could work with a higher output and it would be easier to extrude.

Finally, we know how the samples can be produced in this machine and how it influences the new cooling system in the extrusion of blends; although some of these alternatives should simplify the process significantly or produce the films more easily and faster, in theory.

### 2.3. Background of the project

In order to achieve the best mechanical-optical balance, the idea is to combine LLDPE with Styrene-Butadiene-Styrene (SBS). Styrene-Butadiene Copolymers offer an excellent balance of performance and cost-effectiveness. These copolymers link the gap between high-cost, clear engineering plastics and low-cost, but fragile plastics. More concretely, they present an excellent flexibility, good electrometric properties when cold, abrasion resistance and broad processing range.

SBS rubber is a very common thermoplastic elastomer that is a block copolymer. SBS is made up of a short chain of polystyrene, followed by a long chain of polybutadiene, followed by another short chain of polystyrene. We can appreciate the chemical structure in figure 2.1.

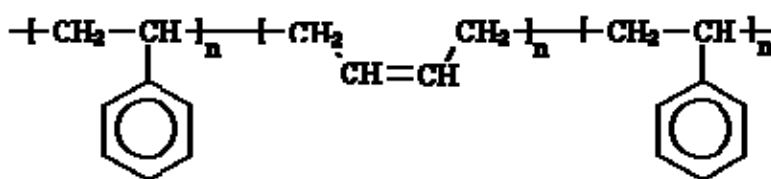


Figure 2.1. Poly(styrene-butadiene-styrene) chemical structure.

If we could stretch out a chain of SBS, it would look like the picture below.



Figure 2.2. SBS chain scheme.

It is known that different polymers do not mix very well, even when they are very similar. This applies to the blocks of SBS just as to any other polymers. So the polystyrene blocks tend to clump together and so do the polybutadiene blocks.

The polystyrene clusters act as crosslink for the polybutadiene blocks and fragment when the SBS is heated, so it can be processed and recycled similarly to a non-crosslinked polymer. It is known that SBS is used as a compatibilizer to improve the properties of some blends. It is very common to combine Polyethylene (PE) with Polystyrene (PS)<sup>(ref. 1)</sup>. The crosslinking occurs between PS and SBS and it has a significant impact on the mechanical properties of the blends. Improvements can be appreciated in terms of impact strength, tensile modulus and the elongation at break. Before, we need to crosslink it partially with a small amount of dicumyl peroxide (DCP) when melting at high temperatures by using.

The residual free radicals in PE will react with butadiene component of SBS at the interface. This article studies the morphology and mechanical properties of PS/PE blends compatibilized by this method. They use PS, LDPE, SBS and DCP.

After comparing the results, the conclusion was that crosslinking between PE and SBS is the key factor which causes the improvement. SBS resins are known for their excellent optical and impact properties and if we can combine them with the appropriate PE or PS we can obtain very good results and a film with an excellent mechanical-optical balance.

Another option is to use SBS to prevent the fracture of PS/HDPE<sup>(ref. 2)</sup>. This article shows how adding SBS, a thin layer covering the HDPE fibers is formed, which improves the PS-HDPE interfacial strength and mechanical properties of the blends. They use PS, HDPE and SBS.

It seems that simply mixing PS with HDPE has a negative impact on the mechanical properties of the blends. If the % in wt/wt of SBS is modified from 10 to 15%, the differences in comparison with the blends without SBS are very significant. The three components were blended together; both ductility and impact strength of the ternary blends were improved considerably.

Based on the results, it is reasonable to propose that the very high toughness found in the PS/HDPE/SBS ternary blends is a result of the synergistic toughening effect of the HDPE and SEBS and the unique microstructure of the ternary blends plays a key role in toughening.

Finally, one interesting patent which combines SBS with different kinds of PE is patent US 7,074,492 B2<sup>(ref. 3)</sup>. The idea of this invention is to demonstrate that the compositions according to Examples 1 to 4, with a structure of A/B/C, make it possible to simultaneously

obtain correct adhesion to PS and EVOH. In this case, SBS is used to study the effect in multilayer films combining metallocene and Ziegler-Natta PE.

The structures combine a coextrusion tie comprising 10 to 35% wt/wt of polymer A with a 40 to 60% wt/wt of SBS block copolymer with 50 to 90 mol% of styrene and 20 to 25% wt/wt of PE. The result is a multilayer structure comprising a layer of tie according to the invention and to the components of comprising such a structure.

Example #1:	(mPE + LLPDE) / SBS / LLDPE	25(50:50)/40/35 %wt/wt
Example #2:	(mPE+ LLPDE) / SBS / LLDPE	25(50:50)/40/35 %wt/wt
Example #3:	(mPE + LLPDE) / SBS / mPE	25(50:50)/40/35 %wt/wt
Example #4:	(mPE + LLPDE) / SBS / LLDPE	15(50:50)/40/35 %wt/wt

These structures can be manufactured by known coextrusion or extrusion blow-moulding processes of thermoplastics technology. Producing the samples using a coextrusion machine is an attractive idea in order to observe the changes in the properties when compared to the monolayer film.

Another remarkable patent is 4,948,641<sup>(ref. 4)</sup>. This patent relates to multiple-layered thermoplastic structures. They are preferably formed by co-extruding or co-injecting a first chemically resistant and non-contaminating inner layer and a second structurally reinforcing outer layer into a blow mold, together with a third layer comprising a copolymer blend and functioning as a tie-layer to bond the mentioned first and second layers.

New multi-layer bottles can be fabricated by blow-molding and provide excellent structural integrity. They can be used to store high purity chemicals which are highly desired by the electronics industry. This multi-layer provides an effective liquid and vapor barrier for storage of high purity and highly reactive chemicals.

The first layer is a chemical resistant and non-contaminating layer fabricated them a fluoropolymer and it is impermeable to the reactive chemicals. The second layer is selected from a groups consisting of PP, PE, polycarbonate, tetrafluoroethylene, polyacrylate and polysulfone. It is a structurally reinforcing layer. And, finally, the third layer is a sandwiched tie-layer, bonding first and second layers. It is a polymer blend comprising polyethylene-vinyl acetate (EVA) and block copolymer of SEBS or SBS with a ratio about 10:90 and a total weight of EVA, SEBS and SBS in the blend of 90:10.

Different combinations can be appreciated. In the first layer the same component, which underwent the swelling test, is always used. It is a very simple, but very useful because it gives an idea of the ability of each monomer to slightly enter into reversible chemical combination with the plastics, hence creating very good adhesion.

In contrast, the second layer can be a blend of 80% Shell Kraton SEBS with a 20% PE or instead of 20% PE it can have a 20% PP. And there is another alternative; it can also be also 80% ethylene vinyl acetate with a 20% shell Chemical.

From this patent, the use of polycarbonate is a good option to improve the mechanical properties of the films, though the opticals worsen. However, it is an interesting option to explore in the project.

To sum up, mixing PE with SBS is possible using another molecule, such as PS, which is not compatible with PE. Here SBS acts as a compatibilizer. Another important thing is the compatibility between PE and SBS. From the literature study a good compatibility is observed. It seems to be that adding SBS in a PE matrix, properties like ductility, impact and tensile strength or elongation-at-break improve significantly. SBS presents excellent optical and good mechanical properties, therefore, in theory, combining it with PE seems to improve the studied balance. However, tensile and secant modulus worsen with the addition of SBS and if the selected SBS is not enough compatible with PE can affect negatively the optical properties. Sometimes a product is required. In one study is mentioned that the addition of DCP is important to improve the PE/SBS compatibility because sometimes can appear an incompatible effects. Components as DCP enhance SBS effect.

This kind of blend has been used in hospitals. The industry produces a flexible material for use in sterilized medical solution container such as blood bags, for example. Other applications are the production of synthetic stoppers or multiple layer containers for storage high purity chemicals.

#### **2.4. Project initial planning**

On the following pages we can see the designed planning of the project to develop it within the specified period of time. Firstly, in table 2.1 we can see all the identified project points and then, in figure 2.3 the resultant Gantt diagram where it is more visual and the information appears more summarized.

Table 2.1. Project planning.

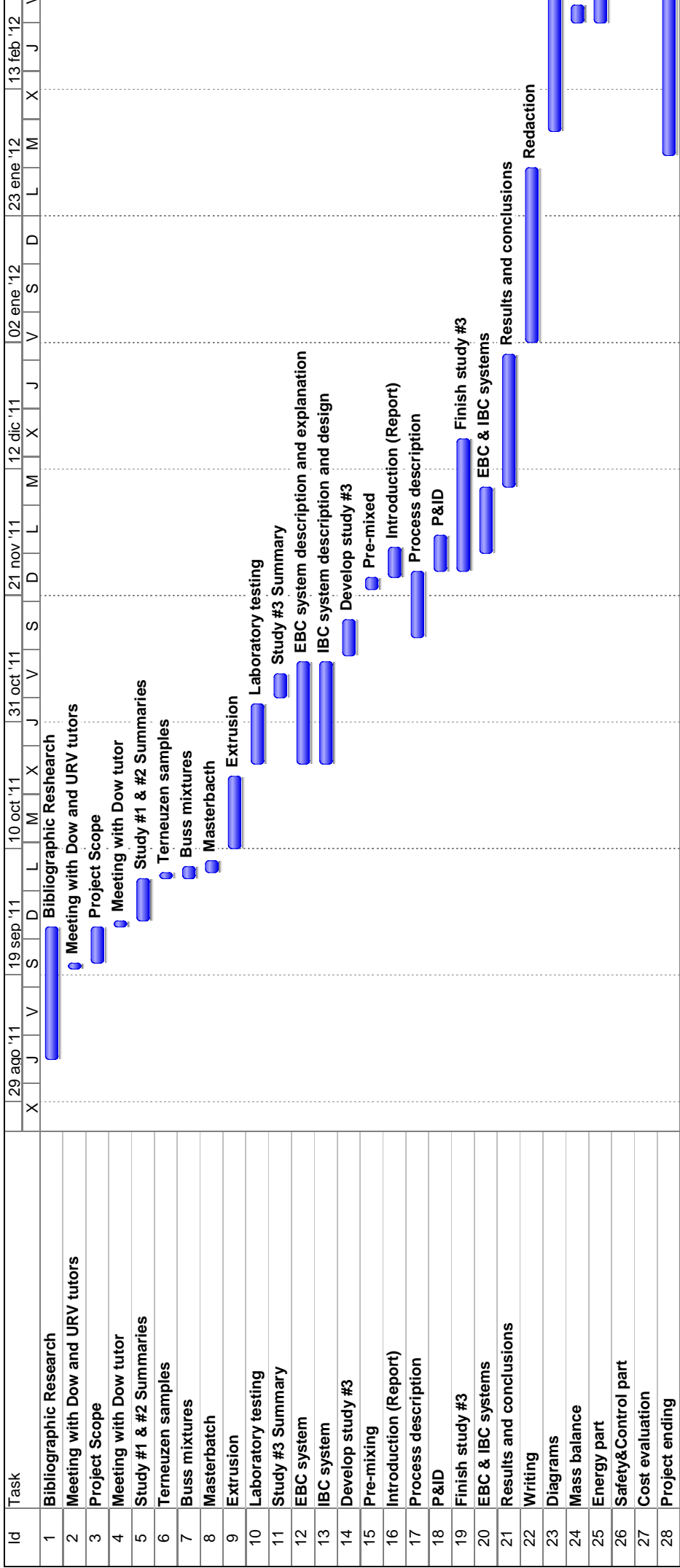
Task	Description	Area	Equipment	Duration	Start date	Completion date
•Bibliographic research	- Recollect information about SBS and SBES in PE blends in articles and patents. Investigate in what kind of products SBS can be blended.	Tarragona TS&D Office	-	16 days	05/09/2011	26/09/2011
•Meeting with Dow and URV tutors	- Define the project scope and the possible parts to study and design.	Tarragona TS&D Office	-	1 day	20/09/2011	20/09/2011
•Project Scope	- Redact the project scope.	Tarragona TS&D Office	-	4 days	21/09/2011	26/09/2011
•Meeting with Dow tutor	- Comment the first study to develop.	Tarragona TS&D Office	-	1 day	27/09/2011	27/09/2011
•Study #1 & #2	- Elaborate the study summaries and check the products availability in the store.	Tarragona TS&D Office	-	5 days	28/09/2011	04/10/2011
•Summaries	- Take a pure sample of each product to send to laboratories in Terneuzen.	Tarragona TS&D Fabrication Lab-G	-	1 day	05/10/2011	05/10/2011
•Terneuzen samples	- Calculate and prepare the mixtures to put in the Buss.	Tarragona TS&D Fabrication Lab-G	Scale	2 days	05/10/2011	06/10/2011
•Buss mixtures	- Prepare the masterbatch PE-SBS to extrude.	Tarragona TS&D Fabrication Lab-G	Compounding - Buss	2 days	06/10/2011	07/10/2011
•Masterbatch	- Dilute the masterbatch to the adequate proportion to extrude the different study samples.	Tarragona TS&D Fabrication Lab-G	Coextrusion Blown (Monolayer Collin)	10 days	10/10/2011	21/10/2011
•Extrusion	- Do all the tests which are specified in the summary.	Tarragona TS&D Lab-G	Tests	8 days	24/10/2011	02/11/2011
•Laboratory testing	- Introduce the results in the database and make conclusion to define the study #3. Elaborate the third study summary.	Tarragona TS&D Office	-	2 days	04/11/2011	07/11/2011
•Study #3 Summary	- Analyze in the extrusion process (Collin).	Tarragona TS&D Department / Out of office	-	13 days	24/10/2011	09/11/2011
•EBC system	-Analyze in the extrusion process (Collin).	Tarragona TS&D Department / Out of office	-	13 days	24/10/2011	09/11/2011
•IBC system						

Table 2.1. Project planning (continuation).

Task	Description	Area	Equipment	Duration	Start date	Completion date
•Develop study #3	- Calculate and prepare the samples, corroborate the products availability, discuss the extrude conditions and define high and very high output.	Tarragona TS&D Department	-	4 days	11/11/2011	16/11/2011
•Pre-mixed	- Pre-mixed the layer B of the samples (study #3).	Tarragona TS&D Fabrication Lab-G	Compounding - Buss	2 days	22/11/2011	23/11/2011
• Introduction (Report)	- Search information and summarize it writing the first part of the report.	Tarragona TS&D Department / Out of office	-	3 days	24/11/2011	28/11/2011
•Process description	- Describe the extrusion process.	Tarragona TS&D Department / Out of office	-	9 days	14/11/2011	24/11/2011
•P&ID	- Extrusion process.	Tarragona TS&D Department / Out of office	-	4 days	25/11/2011	30/11/2011
•Finish study #3	- Extrude and test the samples. Analyze the cooling capacity of the machine system and bring the machine to the output upper limit.	Tarragona TS&D Department	Coextrusion Blown (Collin) and tests	16 days	25/11/2011	16/12/2011
•EBC & IBC systems	- In the Collin. Gather and analyze data of the extrusion process.	Tarragona TS&D Department / Out of office	-	9 days	28/11/2011	08/12/2011
•Results and conclusions	- Take and discuss the results and make conclusions.	Tarragona TS&D Department	-	16 days	09/12/2011	30/12/2011
•Redaction	- Organize all the information and write the report well-structured.	Out of office	-	21 days	02/01/2012	30/01/2012
•Diagrams	- Finish the diagrams and print in the correct format.	Out of office	-	57 days	06/02/2012	23/04/2012
•Mass balance	- Extrusion process.	Tarragona TS&D Department / Out of office	-	2 days	24/02/2012	26/02/2012
•Energy part	- Extrusion process.	Tarragona TS&D Department / Out of office	-	4 days	24/02/2012	28/02/2012

Table 2.1. Project planning (continuation).

Task	Description	Area	Equipment	Duration	Start date	Completion date
•Safety & Control part	- In the Collin. Extrusion process and installations in general.	Tarragona TS&D Department / Out of office	-	32 days	09/03/2011	23/04/2011
•Cost evaluation	- How much the implemented systems cost.	Tarragona TS&D Department / Out of office	-	12 days	23/04/2012	08/05/2012
•Project ending	- Write and check the report continuously.	Out of office	-	72 days	02/02/2012	10/05/2012



### 3. PROJECT DEVELOPMENT BASIS

#### 3.1. Design basis

These sections contain the essential information relating to the project development.

##### 3.1.1. Feed specifications

In the following included information we find the feed parameter. Both tables show the flows expressed in kg/h, but to obtain each sample about 40 - 45 minutes have been required. We need to take into account that this is a semi-batch process.

**Study#2** - It is composted by monolayer samples which have been mixed using a Buss compounder, apart from one sample, sample 7. In order to simplify the mixing operation and to be more time-effective four masterbatches 50:50 were manufactured. They were diluted adding manually the corresponding necessary LLDPE A. The Buss Compounder feed conditions are shown in the following table:

**Table 3.1.** Buss Compounder feed conditions (Study #2).

Masterbatch	Resin base	SBS	% LLDPE A	% SBS	Output (kg/h)	Mass (kg)
A	LLDPE A	SBS 1	50%	50%	15.0	20.0
B	LLDPE B	SBS 1	50%	50%	10.0	5.6
C	LLDPE A	SBS 2	50%	50%	10.0	5.9
D	LLDPE B	SBS 2	50%	50%	10.0	5.9

**Table 3.2.** Blown Extrusion feed conditions in Collin line (Study #2).

Sample	Composition		Output (kg/h)	Mass fraction			
	LLDPE	SBS		LLDPE	SBS	LDPE	PE-g-MAH
1	LLDPE A	SBS 1	4.0	0.85	0.15	-	-
2	LLDPE B	SBS 1	4.0	0.85	0.15	-	-
3	LLDPE A	SBS 1	4.0	0.95	0.05	-	-
4	LLDPE B	SBS 1	4.0	0.95	0.05	-	-
5	LLDPE A	SBS 1	4.0	0.85	0.15	-	-
6	LLDPE A	SBS 1	7.0	0.85	0.15	-	-
7	LLDPE A	SBS 1	4.0	0.85	0.15	-	-
8	LLDPE A	SBS 1	4.0	0.83	0.15	-	0.02
9	LLDPE A	SBS 1	4.0	0.68	0.15	0.17	-
10	LLDPE A	SBS 1	4.0	0.85	0.15	-	-
11	LLDPE A	SBS 2	4.0	0.85	0.15	-	-
12	LLDPE B	SBS 2	4.0	0.85	0.15	-	-
13	LLDPE A	SBS 2	4.0	0.95	0.05	-	-
14	LLDPE B	SBS 2	4.0	0.95	0.05	-	-
15	LLDPE A	-	4.0	1.00	-	-	-
16	LLDPE B	-	4.0	1.00	-	-	-

**Study #3** - The samples of this study are multilayer samples and their structure is the following: A/B/A with 15/70/15 (% in weight).

**Table 3.3.** Study #3 feed conditions (Layers A, 15% each).

Sample	Output (kg/h)	Mass fraction	
		LLDPE A	LDPE
1	1.95	0.80	0.20
2	1.95	0.80	0.20
3	1.95	0.80	0.20
4	1.95	0.80	0.20
5	1.95	0.80	0.20
6	1.95	0.80	0.20
7	1.95	0.80	0.20
8	1.95	0.80	0.20

Layer B includes both 70% of the sample and other components. We decided to feed them in the Buss Compounder to mix them and generate pellets with all the components (output of 15 kg/h). Then these pellets were introduced into the Coextrusion Collin line operating with an output of 11 kg/h.

**Table 3.4.** Study #3 feed conditions (Layer B, 70%).

Samples	Output (kg)	Mass fraction				
		LLDPE A	LDPE	SBS 1	SBS 2	CaCO <sub>3</sub>
1	9.10	0.80	0.20	-	-	-
2	9.10	0.95	-	0.05	-	-
3	9.10	0.95	-	-	0.05	-
4	9.10	0.85	-	0.15	-	-
5	9.10	0.95	-	0.05	-	-
6	9.10	0.75	-	0.05	-	0.20
7	9.10	0.75	-	-	0.05	0.20
8	9.10	0.65	-	0.15	-	0.20

### **3.1.2. Capacity, manufacture flexibility and service factor**

#### **3.1.2.1. Buss Compounder**

The Buss Compounder allows to feed a mix of pellets, which are composed by a portion of each product, and collect them. The machine can operate at 20 kg/h at most. More than 20 kg/h would be working in a risk state and if revolutions increased more and more the kneader or the shell and support devices could be broken. It also requires a minimum output of 1 kg/h, unless sometimes do not reach the desired bubble stability.

However, the most important thing is to select an appropriate temperature profile for the material/s to be extruded, otherwise the screw can block or/and bend.

### **3.1.2.2. Blown Extrusion Collin Line**

This extrusion line is significantly smaller than blown coextrusion line. It is designed to produce monolayer films. In this case, the output is limited by the amperage and pressure. It is necessary to work above 10A and 300bar. These two parameters are directly proportional to the output because the amperage consumption and pressure are linked with it. If the output increases the parameters increase too, and vice versa. One option is to move the temperature profile up to a higher profile, but it difficult exceed 10 kg/h without surpassing the safety indications. However, with an output of 10 kg/h we can extrude the material, but it is not possible to maintain a stable bubble because the refrigeration system cannot cool enough. Then, we need to work with outputs below this. Also, we need to take into account that there is a minimum output. It depends on the sample's width. The wider the sample is, the higher the output must be, unless the bubble remains unstable or breaks. In this case a film of 23.5 cm is required. When working below 2.5 – 3.0 kg/h results is impossible to extrude a film. To ensure a degree of quality the output must be over 3.5 kg/h.

Another limitation is the thickness distribution as the machine does not provide a good one. Depending on the extruded materials, the distribution is not good and it exceeds the limit (10%). Then it is necessary to check the samples and select the appropriate parts to test.

### **3.1.2.3. Blown Coextrusion Collin Line**

This coextrusion line was designed to work at 30 kg/h at most, but the machine can only reach 12 - 15 kg/h because it is limited by the refrigeration. The film cannot cool so fast and if we increase the output the bubble becomes unstable and breaks.

These are the top limitations of this machine. Therefore, in section 4.3 different alternatives are suggested to move the output upwards to more than 12 - 15 kg/h. At the same time, we have a lower limit. The more layers you extrude the higher output you require. Unless the bubble is unstable because it is more difficult to refrigerate. A three-layer film is not possible to extrude below 5 kg/h in the study conditions.

This line allows to extrude films from 1 to 7 layers, one of which with 3 different components. However, you can elaborate a mix and introduce it into the hopper. Each extruder has a refilling system which provides a 5 kg load within a few seconds. This system

is the key to transform this line into an effective semi-batch process. When the extrude alarm rings, the system refills the hopper through the tubes which are inside the material sacks. This process can be manual or automatic; the only condition is that it is always indispensable to pay attention to the material levels in the sacks.

Finally, the three mentioned machines are a semi-continuous process so that is possible to switch on and off the machines every day. Indeed, this is the operation process, but when the workday finish the machines can be turned off. It must be taken into account that the department is an investigation department and its main purpose is not to produce and sell. The valuable goal is to produce enough quantity to test the samples and provide study requestors or customers with information about the samples and the process in order to improve it or take conclusions. Therefore, there is no specific service factor. The machines to be used depend on the number and size of the studies which have been requested.

### 3.1.3. Product specifications

All the samples must fulfill the following parameters:

**Table 3.5.** Product specification.

Parameters	Specification	Comments
Thickness ( $\mu\text{m}$ )	50.0	Good distribution
Width (cm)	23.5	-
Orientation degree	High	-

### 3.1.4. Raw materials and products conditions

In the following tables the conditions of the raw materials and products throughout the process are showing in the following tables. It must be taken into account that the Buss products feed the extrusion lines.

**Table 3.6.** Buss Compounder temperature profile.

Study	Line	LAYER B Sample	Temperature profile ( $^{\circ}\text{C}$ )				
			$T_{\text{SCREW}}$	$T_{\text{HEATER 1}}$	$T_{\text{HEATER 2}}$	$T_{\text{HEATER 3}}$	$T_{\text{DIE}}$
#2	Buss Compounder	All	110	125	140	135	105
		2	110	125	140	135	105
		3	115	200	195	190	100
		4	110	125	140	135	100
#3	Buss Compounder	5	110	125	140	135	115
		6	115	200	195	190	100
		7	115	200	195	190	100
		8	115	200	195	190	100

**Table 3.7.** Buss Compounder process conditions.

Study	Line	Sample	Kneader Speed (rpm)	Screw speed (rpm)	Die gap holes
#2	Buss Compounder	A	160	105	7
		B	120	75	7
		C	120	75	7
		D	120	120	7
		2 (layer B)	120	75	7
		3 (layer B)	120	75	7
		4 (layer B)	120	75	7
		5 (layer B)	120	75	7
#3	Buss Compounder	6 (layer B)	120	75	7
		7 (layer B)	120	75	7
		8 (layer B)	120	75	7
		8 (layer B)	120	75	7

**Table 3.8.** Collin's temperature profiles.

Study	Line	Sample	Layer	Temperature profile (°C)					T <sub>DIE</sub> (°C)	
				T1	T2	T3	T4	T5	T <sub>DIE 1</sub>	T <sub>DIE 2</sub>
				SCREW	SCREW	SCREW	SCREW	SCREW		
#2	Extrusion Collin	1	A	190	225	235	240	242	240	240
		2	A	190	225	235	237	240	242	242
		3	A	190	225	235	240	240	240	240
		4	A	190	225	235	237	240	242	242
		5	A	190	225	235	240	241	240	241
		6	A	190	230	245	255	251	244	242
		7	A	180	207	216	221	225	212	212
		8	A	190	225	235	239	240	242	242
		9	A	190	226	235	238	240	243	242
		10	A	195	222	229	234	239	231	229
		11	A	180	207	218	220	223	212	212
		12	A	190	225	235	237	240	242	242
		13	A	190	225	235	240	241	240	241
		14	A	190	225	235	237	240	242	242
		15	A	190	225	237	243	240	240	240
		16	A	190	225	235	237	240	242	242

**Table 3.9.** Collin's temperature profiles.

Study	Line	Sample	Layer	T1	T2	T3	T4	T5	T5	T <sub>DIE</sub>
				SCREW	SCREW	SCREW	SCREW	SCREW	SCREW	
#3	Coextrusion Collin	1	A	179	220	231	237	235	235	240
			B	190	235	245	245	250	-	239
			A	180	220	230	235	235	-	240
		2	A	180	220	230	237	235	235	240
			B	200	240	241	248	250	-	240
			A	179	220	230	235	235	-	240
		3	A	180	220	230	235	236	235	240
			B	201	240	245	251	250	-	240
			A	180	220	230	235	235	-	240

**Table 3.9.** Collin's temperature profiles (continuation).

Study	Line	Sample	Layer	T1	T2	T3	T4	T5	T5	T <sub>DIE</sub>
				SCREW	SCREW	SCREW	SCREW	SCREW	SCREW	
#3	Coextrusion Collin	4	A	180	220	230	235	234	235	240
			B	200	240	242	251	250	-	240
		5	A	180	220	230	235	235	-	240
			B	180	220	230	235	236	235	240
			A	200	240	245	245	250	-	240
		6	A	180	220	230	235	235	-	240
			B	201	240	243	244	250	-	240
			A	180	220	230	235	235	-	240
		7	A	181	220	230	235	235	235	240
			B	201	239	241	245	250	-	240
			A	180	220	230	235	235	-	240
		8	A	180	220	230	235	236	235	240
			B	200	240	242	246	250	-	240
			A	180	220	230	235	235	-	240

**Table 3.10.** Collin's process conditions.

Study	Line	Sample	Layer	Melt temperature (°C)	Melt pressure (bar)	Screw speed (rpm)	Take-off (m/min)	Die gap (mm)	B.U.R. (:1)
#2	Extrusion Collin	1	A	239	142	45	3.3	0.8	2.5
		2	A	239	88	45	3.7	0.8	2.5
		3	A	239	170	45	3.3	0.8	2.5
		4	A	239	94	45	3.7	0.8	2.5
		5	A	239	168	45	3.3	0.8	2.5
		6	A	251	170	45	4.9	0.8	2.5
		7	A	224	187	45	3.5	0.8	2.5
		8	A	239	154	45	3.4	0.8	2.5
		9	A	239	136	45	3.7	0.8	2.5
		10	A	238	157	45	3.6	0.8	2.5
		11	A	225	190	45	3.3	0.8	2.5
		12	A	239	89	45	3.7	0.8	2.5
		13	A	239	168	45	3.3	0.8	2.5
		14	A	239	93	45	3.7	0.8	2.5
		15	A	239	181	45	3.3	0.8	2.5
		16	A	239	97	45	3.7	0.8	2.5

**Table 3.10.** Collin's process conditions (continuation).

Study	Line	Sample	Layer	Melt temperature (°C)	Melt pressure (bar)	Screw speed (rpm)	Take-off (m/min)	Die gap (mm)	B.U.R. (:1)
#3	Coextrusion Collin	1	A	235	171	32	9.1	1.8	2.5
			B	242	274	87			
			A	217	162	34			
		2	A	228	156	32	8.0	1.8	2.5
			B	243	283	87			
			A	216	154	35			
		3	A	227	156	32	8.2	1.8	2.5
			B	243	283	87			
			A	216	154	35			
		4	A	227	157	32	8.2	1.8	2.5
			B	244	253	87			
			A	216	153	35			
		5	A	227	156	32	8.2	1.8	2.5
			B	242	280	87			
			A	216	153	35			
		6	A	228	157	32	8.1	1.8	2.5
			B	242	291	87			
			A	216	153	36			
		7	A	228	159	32	8.0	1.8	2.5
			B	243	292	87			
			A	216	152	35			
		8	A	227	155	32	7.9	1.8	2.5
			B	242	256	87			
			A	217	150	35			

When the raw materials arrive in the department, they are classified and sent to storage. The extruded samples undergo the corresponding tests in the laboratory; therefore, it is only required to move them to the appropriate laboratory trolley with the correct notification on the register.

### **3.2. Basic data to the engineering development**

Sections 3.2 show the basic data to develop the project. Here the site location is specified and the condition of the area which it belongs to.

#### **3.2.1. Available utilities**

It is very important to know which are the plant utilities and the conditions which they are in. Subsequently, they are listed and characterised.

- **Air**

The instrumentation air, which is used in the plant and laboratory, is taken from the atmosphere, then it is dried freezing the water which contains and then it is pressurized at 8

kg/cm<sup>2</sup>. The temperature depends on the environment conditions, but it is usually around 20 - 25 °C

- **Refrigeration water**

The refrigeration or cooling water is served at 20°C and at a pressure of 4 kg/cm<sup>2</sup>. When this water returns its temperature is higher and the pressure has dropped. This water returns to the cycle where it is load at a pressure of 6 kg/cm<sup>2</sup> in order to compensate loses until it reaches the plant. But, before pressurized, it passes through decanters where the water is conditioned to room temperature.

- **Electricity**

There are two electricity feeding lines in the building. The main line supplies electricity and from there, different lines leave to feed the other buildings around. The main line has an intensity of 2000A and it is reduced using electrical transformers or using one of the three phases with the neutral cable (220V). Simultaneously, there are two other machines which have a higher consumption and are fed by another electricity line. These machines are the big coextrusion cast and blown lines.

**Table 3.11.** Electricity used in the building.

Intensity (A)	Voltage (V)	Frequency (Hz)	Phase	Applications
2000	380	50	3-phase	General feed to the building area.
	220	50	1-phase	Building area (conventional plugs).
1000	380	50	3-phase	Big coextrusion cast line.
800	380	50	3-phase	Big coextrusion blown line.
500	380	50	3-phase	Small and medium extrusion and coextrusion lines.
	24	50	3-phase	Instruments.

The building area has the corresponding drains in order to carry off rainwater and/or liquid waste. It is an area where sometimes there are heavy precipitations and, therefore, the drains must be regularly checked.

### **3.2.2. Energy fees**

The different energies used have a specific price in the market. The following values are provided by the PFC coordinator.

**Table 3.12.**

Energy	Unit	Fees (€/unit)
Electricity	MWh	76.00
Gas	MWh	27.00
Dirty water	m <sup>3</sup>	0.63
Demineralized water	m <sup>3</sup>	1.50
Steam 20kg/cm <sup>2</sup>	Tone	21.00
Steam 40kg/cm <sup>2</sup>	Tone	29.00
R134a <sup>1</sup>	kg	≈12.00
Nitrogen	m <sup>3</sup>	0.08

### 3.2.3. Site location

The industrial complex is located on the outskirts of Tarragona (Catalonia), on the northeastern coast of Spain.



Spain.



Industrial site

The facilities are owned and run by Dow Chemical Company, which is based on the production of PE. The complex includes two production areas: north and south. The first

is composed by an Octene plant and Ethylene cracker. In contrast, the south area comprises derivation plants such as LDPE or HDPE plants which manufacture a wide range of products.

The whole site covers an enormous extension of land with a big number of plants and buildings on it. It currently has about 600 employees and there are 8 plants with 11 production lines. Specifically, the plant, referred in the project, belongs to the Technical Service and Development (TS&D) department, which works on the study of polymers.

The TS&D department, located in Tarragona South, provides international services and is one of the most important centers in Europe. Apart from studying the polymer and their science; it is focuses on customer service. The department develops new blends in different conditions fixed by the customers and provides them with feedback.

<sup>1</sup> <http://www.eurorefrigerant.com/lang-es/>

**Table 3.13.** Basic information of Dow Tarragona.

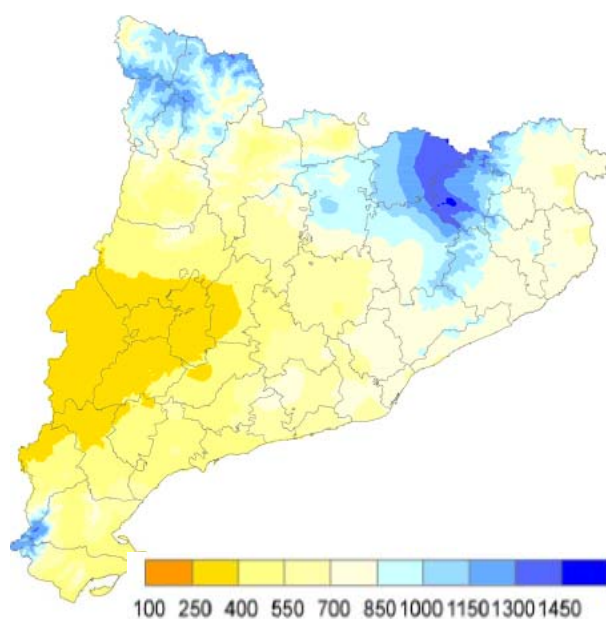
<b>Country</b>	Spain	<b>Altitude (masl)</b>	68
<b>Community</b>	Catalunya	<b>Location</b>	41,100225°N and 1,1855928°E
<b>Province</b>	Tarragona	<b>Surface (km<sup>2</sup>)</b>	62,35
<b>Region</b>	Tarragonès	<b>Population (hab. 2011)</b>	134.085

### 3.2.3.1. Environmental conditions

The plant is located in Catalonia where the climate is generally Mediterranean. Tarragona area is situated in the Mediterranean coast. The main characteristic of the coast Mediterranean climate is its mild winters.

The presence of ice is not common because temperatures are usually not below 0°C and the average temperature in December, January and February is around 9°C. In contrast, summer temperatures lay around 24-25°C with a high environmental humidity. It is very uncommon register a temperature above 27-29°C. The relative humidity is about 70-75% and on the littoral it is nearly homogeneous throughout the year.

The average precipitations during the year in Tarragona rounds 500 mm. However, heavy rain may fall in autumn.



One remarkable aspect is that rain in this kind of climate are very irregulars.

The figure shows the distribution of the rainfalls in Catalonia. It can be appreciated that the highest values are concentrated in the main mountainous reliefs. In contrast, the area of Tarragona presents medium-low values. In general, the south of Catalonia is not an important precipitation area in comparison with the north and northeast.

**Figure 3.3.** Geographical distribution of the precipitations in mm (Sep. 2010 to Aug. 2011)<sup>(ref. 5)</sup>.

The following figure shows the tendency throughout the year. There are represented the temperature and precipitation average values which are the result of the period 1971-2000. It is more representative than the database of one specific year.

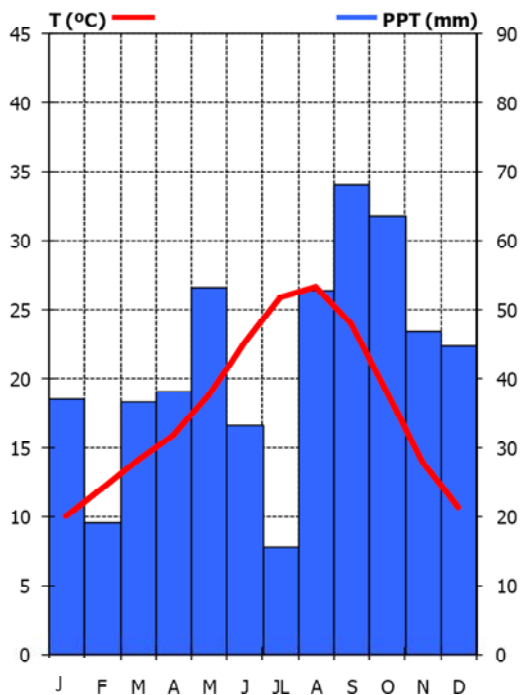


Figure 3.4. Tarragonès climatology (1971-2000).<sup>(ref 6)</sup>

It can be observed that the distribution of the precipitation is irregular. The values correspond to the register values of all the region of Tarragonès. For this reason, the maximum precipitation value is below to the average of Tarragona which is mentioned above. Rainfalls are more generous in autumn and the less in summer. In terms of temperature, the figure shows what it is mentioned above: hot summers and moderate winters. Only in winter can be appreciated the presence of ice, but it is not common.

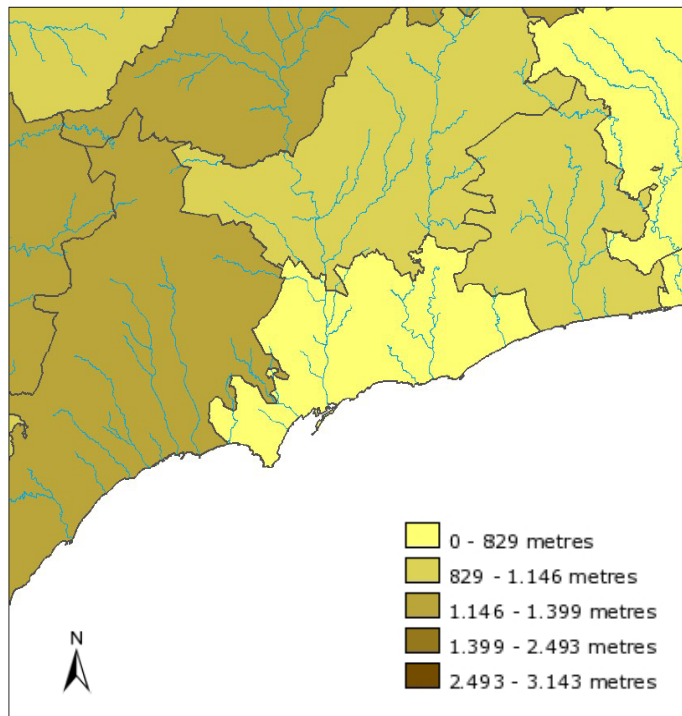
The following table contains the general climatological information of Tarragona. The values are annual averages.

Table 3.14. Meteorological information, Tarragona (2011)<sup>(ref 7)</sup>.

CLIMATOLOGY PARAMETERS	VALUE
Total Precipitation (mm)	593,2
Average temperature (°C)	17,2
Maximum average temperature (°C)	30,0
Minimum average temperature (°C)	2,4
Maximum absolute temperature (°C)	33,6
Minimum absolute temperature (°C)	-7,4
Average pressure (mbar)	1008,6
Maximum absolute pressure(mbar)	1027,7
Minimum absolute pressure (mbar)	983,8
Windspeed (m/s)	2,8
Humidity (%)	72,5
Global average radiation (MJ/m <sup>2</sup> )	6,94

### 3.2.3.2. Ground conditions

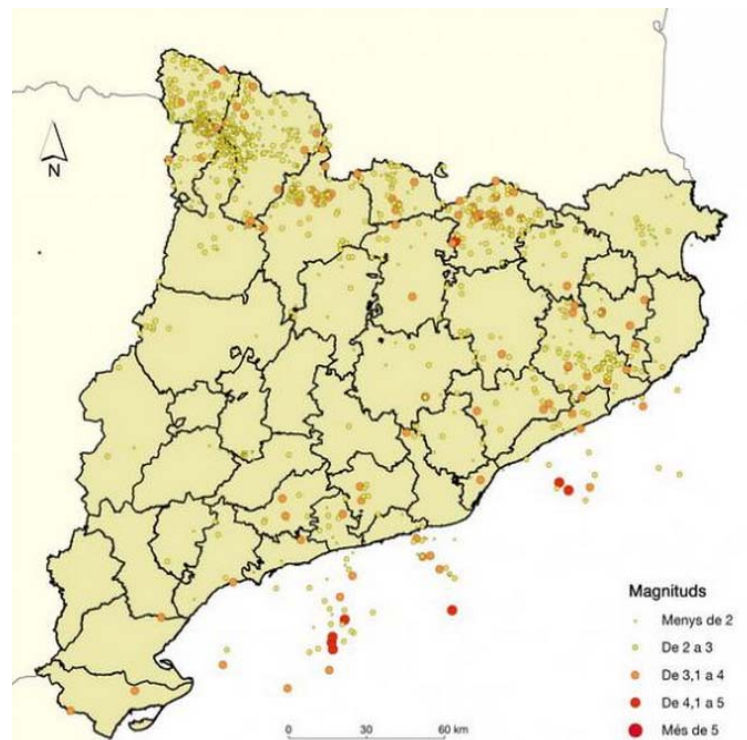
The area presents a very homogeneous relief. Figure 3.6 shows the altitude of the area and figure 3.7 the recorded seismic activity during the period from 1977-1997.



**Figure 3.5.** Tarragonès geography<sup>(ref. 8)</sup>.

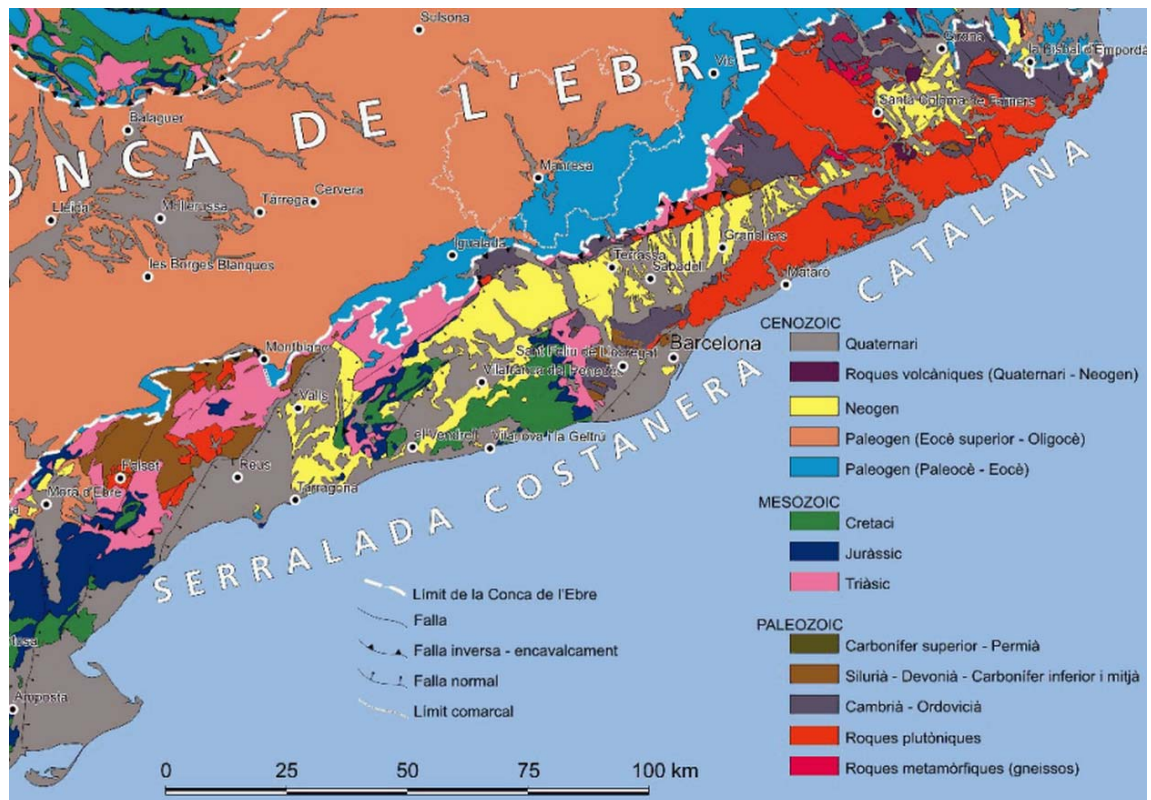
As the figure shows, the area around Tarragona does not present an aggressive relief with high altitude differences; homogeneity dominates. The region is found below 829 meters, which is normal taking into account its location next to the sea. Another important factor to be considered is the seismic action. If the plant is located in a place where the presence of earthquakes is common it is not the same as in a place where they are unusual.

As it can be observed in the figure above, Catalonia is not an important place to take into account in terms of earthquakes. The highest ones registered occurred in the sea, especially in the northwestern area. In Tarragona there are no significant movements and the most important occurred 30 km off the coast.



**Figure 3.6.** Catalonia's seismic map<sup>(ref. 8)</sup>.

Finally, a figure is presented to show the geology of the area where the plant is located. On the right of the map, there is a color codification so as to distinguish the soil components.



**Figure 3.7.** Geological map of the Mediterranean coast (ref. 9).

It is basically composed by Cenozoic soil (Quaternary and Neogen). Although is nearly all Quaternary. This kind of soil is characterized by contain sands, clays and silts.

### 3.2.4. Official design rules and codes

Designing and carrying out the test procedures must follow the codes and normative:

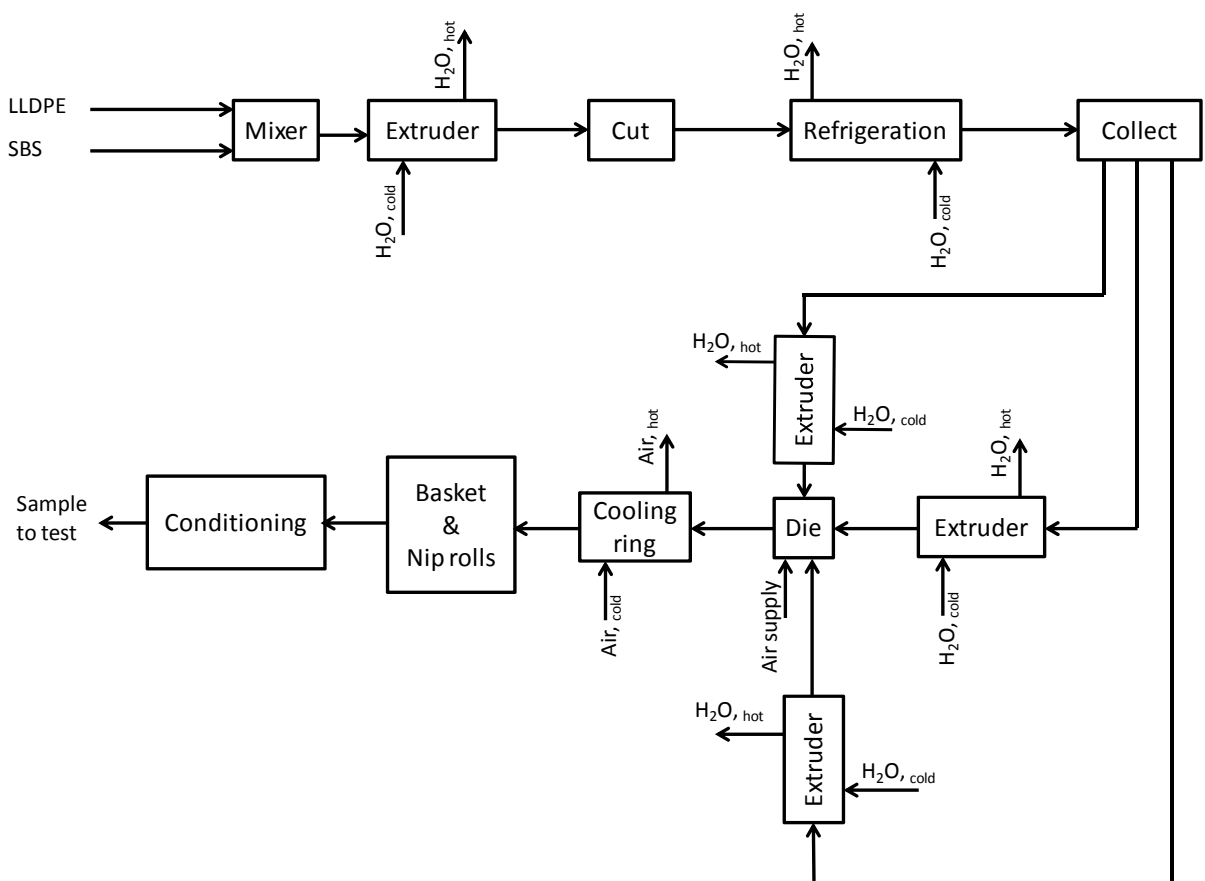
- API 610 for pumps.
- Dart drop impact test based on ISO 7765-1/1988.
- Elmendorf test follow ASTM 1922.
- Seal strength test based on ASTM F2029-00.
- Hot tack test follow ASTM F-1921.
- Puncture test based on ASTM D 5748-95 (2007).
- Gloss, Clarity and Haze follow ASTM D-2457-90, ASTM D1746 and ISO 14782, respectively.
- Oxygen transmission test based on ASTM D-3985.

## 4. BASIC ENGINEERING DEVELOPMENT

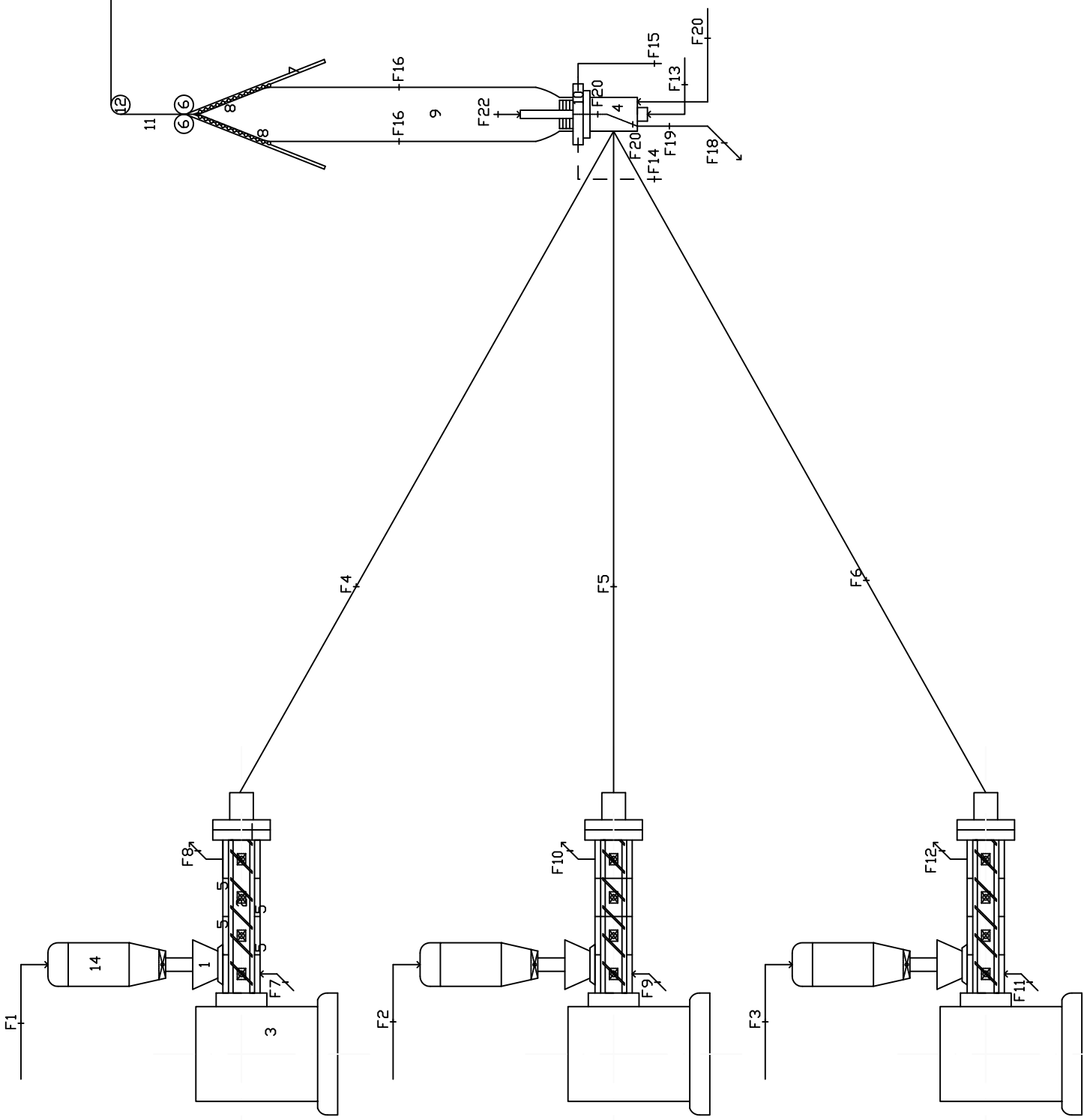
### 4.1. Diagrams

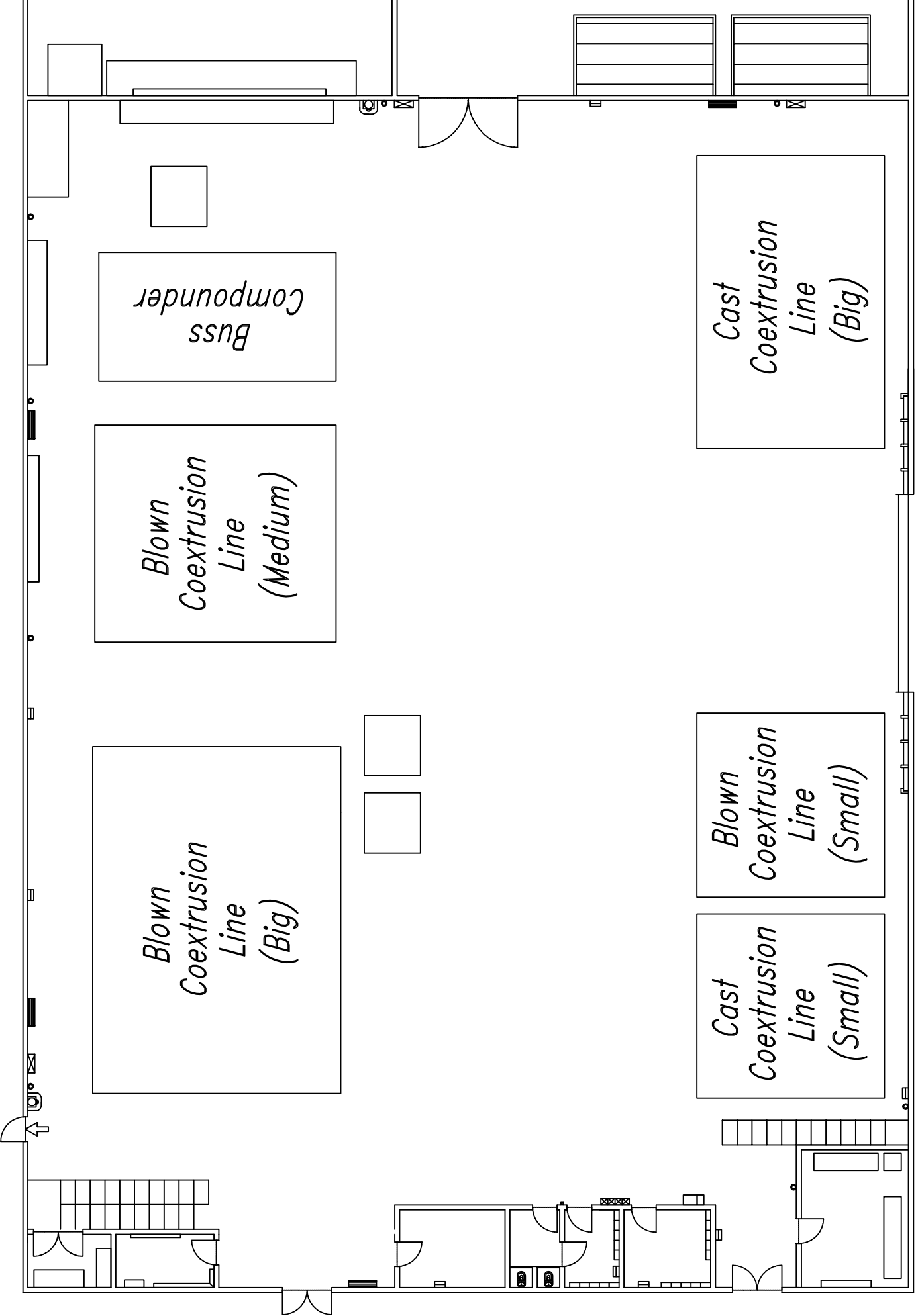
#### 4.1.1. Conceptual block-diagram

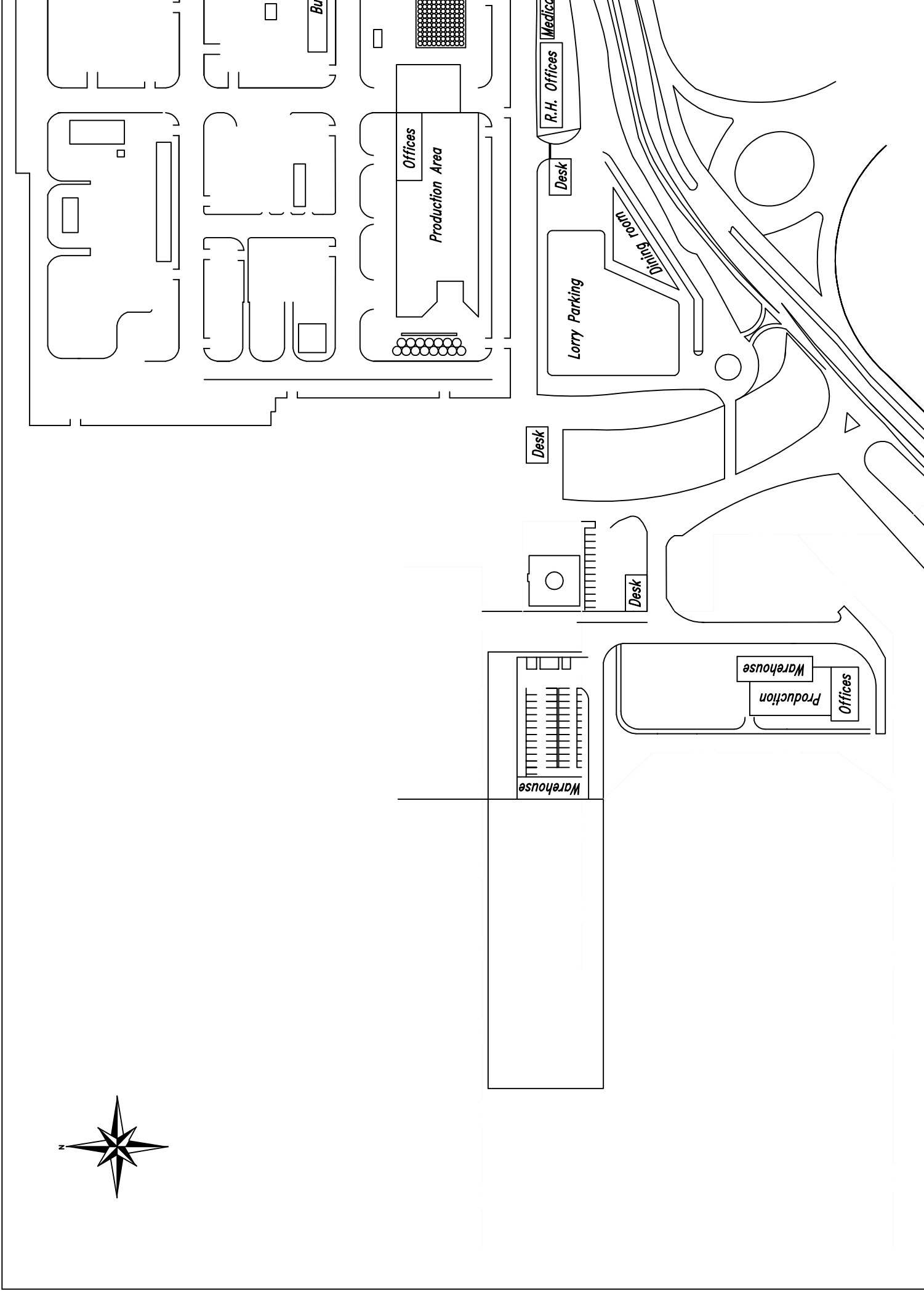
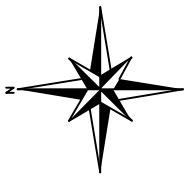
The process to manufacture the multilayer blown studied films can be understood as a semi-continuous process with previous treatments. First of all, LLDPE and SBS are introduced in a mixer where are mixed about 20 minutes. Then the mix is introduced into the Buss compounder where the mix is melted, compressed, cut and refrigerated with cold water. The resulting pellets are collected and introduced into the multilayer blown Collin line. In this case there are three inlets because the films must have three layers. Here, the pellets are melted and compressed. Then the resulting material are swollen and refrigerated with air when it passes through the die and cooling ring. Finally the film is collected in a roll, using a basket and nip rolls, and it is conditioned to test in the laboratory.

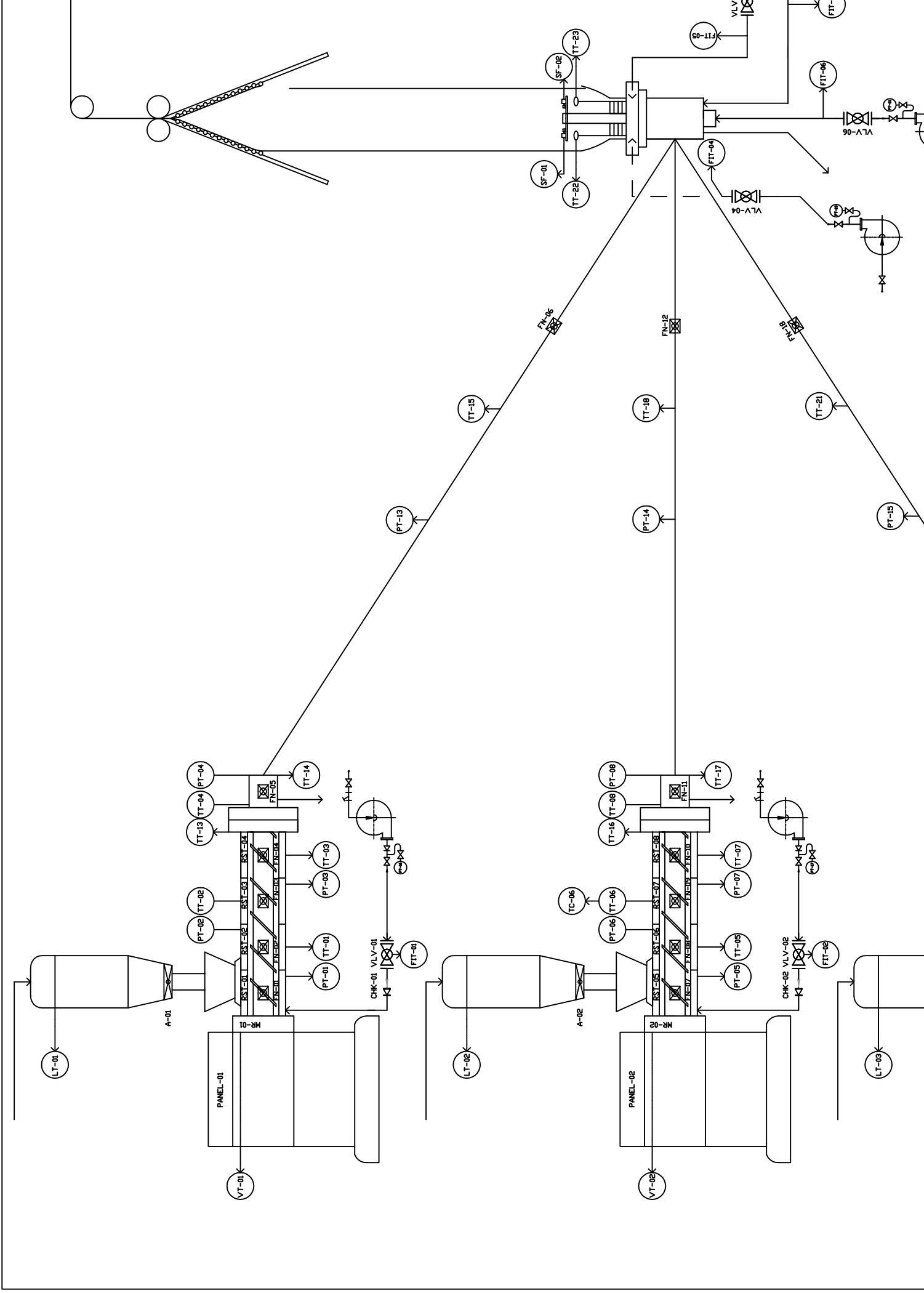


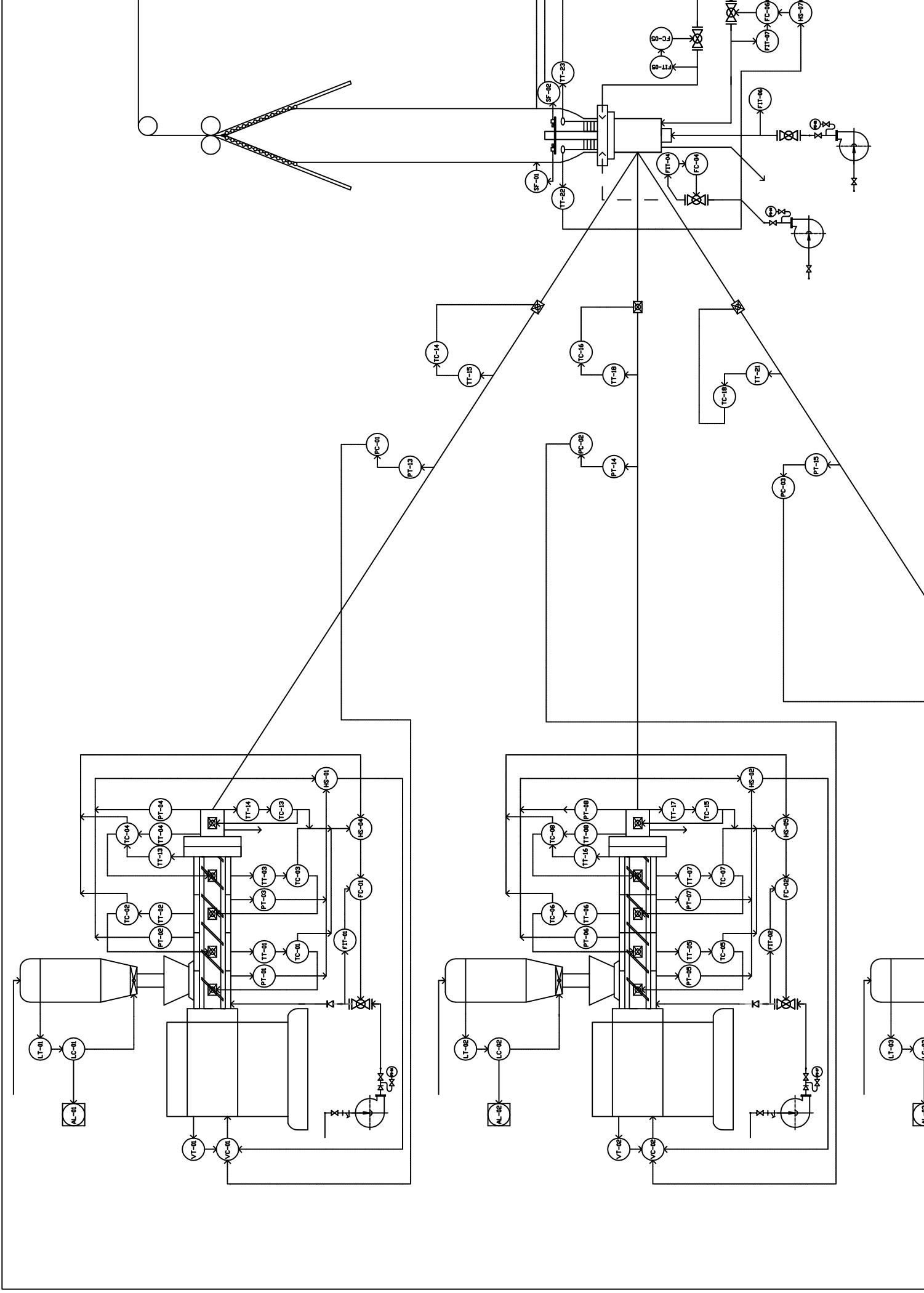
**Figure 4.1.** Conceptual block-diagram of the multilayer film production.











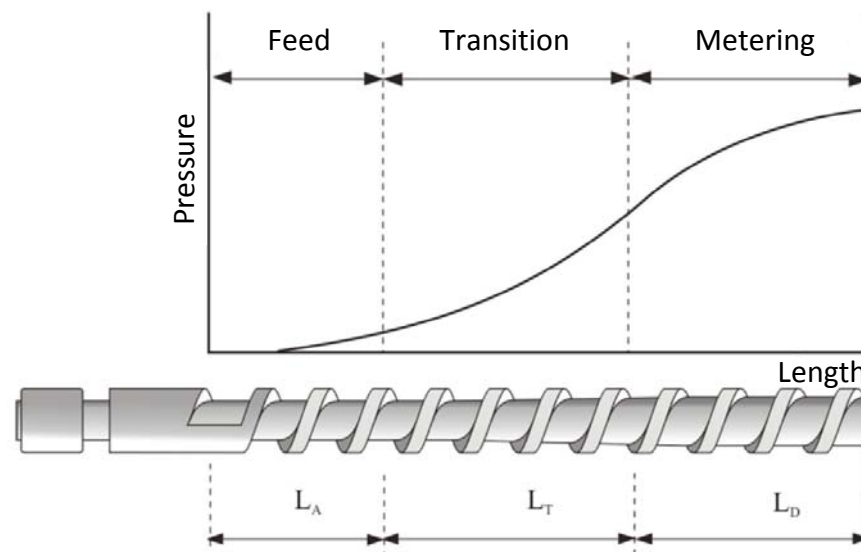
## 4.2. Descriptions

### 4.2.1. Process description<sup>2</sup>

The extrusion process consists of pushing the material, such as polyethylene, through a die of the desired cross-section, which is a specialized tool used in manufacturing industries to cut or shape material using a press. By means of this process it is possible to create complex cross-sections and work materials that are fragile. This process is a coextrusion process which uses three extruders in order to manufacture a three-layer film. The process is the same in every extruder.

The polymer is fed into the hopper **1**, and then the screw **2** mixes and pushes the pellets along the extruder **3** where they are melted and pressurized. Moreover, this line includes a 5kg-capacity-refill system **14** which offers the possibility of always having 5 kg in the hopper and simulating a continuous process.

Three different regions can be distinguished in the screw: feeding, transition and metering. These are shown in figure 4.2.



**Figure 4.2.** Extruder regions.

The feeding region presents the highest channel depth due to its location next to the hopper where the polymer is still completely solid. In this region the pellets are compacted and transported to the transition region. In this second region, the intermediate part of the extruder and the channel depth decreases gradually. The solid pellets are more pressurized in order to compact more. Then the solid mass is melted and propelled to the next region,

<sup>2</sup> This section includes reference numbers in order to understand better the process drawn in section 4.1.2.

the metering region. In this last part, the extruder presents a very low channel depth because it is located next to the die **4**. The melted polymer mass is homogenized and more pressurized so as to apply more strength to push it into the die.

Each screw has a cooling system with cold water to refrigerate the extruder and reduce the temperature and a heating system with electrical resistances **5** so as to increase it. These systems are very important because the extruder must have the possibility to heat or cool the polymeric mass which usually melts at about hundreds of degrees and if some problem occurs and the extruder temperature drops dramatically, the screw could block and break when it runs again. For this reason, an accuracy control of the temperature is required to heat or cool the polymer whenever necessary. It is very common to use three independent heating regions to reach the appropriate temperature gradient along the extruder.

During the extrusion, the polymer is pressurized. When it is introduced into the hopper, the pellets are at atmospheric pressure and from this point, the melt pressure in the die increases up to 150 – 290 bar. The channel through which the polymer passes does not have the same section from beginning to end, as we mentioned. The channel is much narrower at the end because there more pressure is required to push the polymer to the die where it takes form.

When the melted polymer arrives to the die it is molded. This is a blown process with a circular die. The polymeric mass leaves through the die forming circles of polymer. At this point, this mass is pulled by two nip rolls **6** whose speed can be modified depending on the output or desirable thickness. While the nip rolls are pulling, a basket **7** composed of small rolls **8** helps to avoid creases and folds in order to ensure a good quality film. All of this process is possible because, whilst the polymeric mass is pulled forming a closed cylinder **9**, a cooling ring **10** refrigerates it externally with cold air at 14°C which is sent out. At the end of the basket, nip rolls join both sides of the bubble producing a two-sided laminar film **11**, having each of the sides three layers. Finally, more nip rolls **12** drive the film to the reel **13** where the film is rolled up.

#### **4.2.2. Facilities description**

The industrial complex located on the outskirts of Tarragona is divided in two regions: north and south area. This plant is in the south area, therefore, the plans corresponds on this second part of the site. It includes 8 different derivation plants with 11 production lines.

As the layout plan shows, plan of section 4.1.4, this region has 3 access points along the part located by the motorway. This is important so that employees and carriers can access and leave the site easily.

In the central area next to the motorway, there is the main desk and on its right, the Human Resources (HR) offices. This area includes offices and a presentation room used for presentations and important events. Next to the HR offices there is the medical centre. It is also located next to the motorway as a preventive measure.

Next to the desk, there is a canteen and a big car park. In this south area there is only one canteen for all the employees, however, there is another one in the north area.

In the northeastern area there are different derivative plants that manufacture low and high density polyethylene with the corresponding offices, laboratories and warehouses. This area is focused on producing to satisfy the sales volume. And, the maintenances area is located at the far end of this northeastern area.

In contrast, the left side of the southern area combines production and research. The TS&D department is located at the southwestern end. This department includes two buildings (P and Q). The P building is where the studied plant is located. This department is devoted to the study of polymer science. Its aim is not production, but research and customer services.

The P building has two floors of offices; some are on the ground floor and there is also a test laboratory, a fabrication laboratory and a meeting room. In the fabrication laboratory, which is included in the plot plan section 4.1.3, different kind of extrusion lines can be distinguished. This area has 5 access points, including emergency doors, through two of which a fork-lift truck can drive. There are two small coextrusion lines which produce 200mm-wide films with limit outputs of 10-15 kg/h. One of these two is a cast line, which uses a flat die. In contrast, the other is a blown line and the die is circular. Just next to this last one line there is a big cast coextrusion line. This is the biggest line in the department. It requires to work with high outputs and therefore it is used for reproducing better industrial extrusion conditions. In front of this line there is the Buss compounder used in study #2 which mixes better the different polymers. This line is smaller than the others because it does not require much space. Next to it there are two blown coextrusion lines. Both lines are blown processes, but the machine next to the Buss compounder is the biggest. The other is smaller. It probably requires more space because the blown processes which produce very

wide films (about 1 m) require many meters to refrigerate the bubble so as to join both sides properly.

These are the main machines in the department. Then, there are smaller extruders, one line cast one line blown above the small coextrusion lines, but they are not usually used due to their precision problems.

Finally, on the right part of the plot plan two more areas can be appreciated. The first from left to right includes a small storage and one room where there is some tests equipment. And the part more on the right is an area to keep material, samples and other things. It is important to point out that part of the Q building, next to the P building, is a warehouse where raw materials are stored.

### **4.3. Basic design**

The blown coextrusion Collin line used in order to manufacture the films is designed to work at 30 kg/h. However, the maximum output used is about 12 or 15 kg/h. The limitation is the refrigeration. The bubble cannot be refrigerated as fast as it should so as to increase the output. When it is increased over 12-15 kg/h some instabilities appear.

In order to solve this problem different alternatives are studied. There are three options: install one Internal Bubble Cooling (IBC) system in the line, change the cooling ring and/or pre-cool air with a refrigeration cycle.

The three alternatives have been analyzed so as to know which one can be the best option or if it is appropriate to implement all of them.

#### **4.3.1. Internal bubble cooling system**

The Internal Bubble Cooling system, known as IBC, works by placing a cooling tower next to the die which acts as a heat exchange providing a mean to introduce cold air into the bubble and remove the hot air.

This device provides cold air which is introduced into the die through a channel, next to the die inner wall. At the same time, the hot air is removed through another channel inside of the other one. The designed system introduces cold air from the inferior part of the bubble through lateral holes in the base of the tube. Then the hot air is removed using the outlet tube which is the inlet of hot air within the bubble.

Other old systems use a longer tube which is full of holes and bent at the end. In this case, the diffuser tube is built in order to rotate with the die and it is attached to it <sup>(ref. 10)</sup>.

In contrast, in this case, the tube emerges from the inner part of the die. The cold air, which is sent out through the holes, expands the material beyond the die diameter. The bubble diameter can oscillate between 2 or 3 times the die diameter. The effect of external cold air provided by the cooling ring also contributes to the final expansion.

Depending on the tubes diameter, the flow rate of the internal cooling air is higher or lower. The higher the flow rate is, the higher the internal pressure. This implies the presence of more confined air in the bubble, then there is more expansion and higher bubble diameter.

While the cold air refrigerates the bubble, a slim line can be observed in the bubble. This line is called frost line; it appears as a result of the polymer state change into solid. It is the point beyond the die where the temperature of the molten polymer falls below the softening point and the diameter of the extruded bubble stabilizes. The frost line height depends on the melt temperature, the extrusion and cooling speed and the bubble diameter.

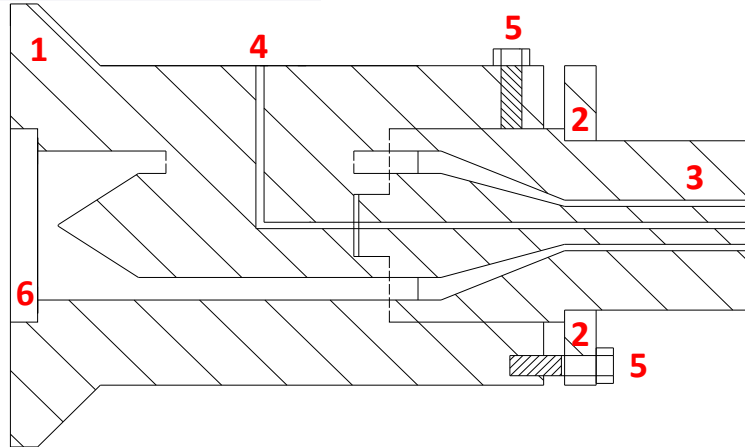
The higher the melt temperature and the lower the cooling speed are, the higher the frost line height is being more difficult to control the thickness film distribution which should be as homogeneous as possible. The reason is that in these conditions, the polymer requires more time to solidify and the resulting film is more transparent and smooth.

The stability also depends on the vertical sensor location with regard to the frost line. The idea is to locate the sensors at or slightly above the frost line in order to avoid possible delays in the signal. These can be due to changes in the shape of the bubble, which can be misinterpreted, or in the required time because of differences in the tube circumference made at the frost line to travel beyond to the sensors.

Installing an IBC system would imply very important improvements, but at the same time, the cost is very high. On the one hand, the implementation such a system in the Collin coextrusion line would imply an improvement in the output which can reach up to a 50% more. Technically it is a very good solution and would satisfy what we are looking for. The idea is very simple, IBC system doubles the cooling area because we are refrigerating the bubble both externally and internally and, therefore, this implementation would provide stability to the bubble and, as a result, the rollers can increase their pulling speed and the line production capacity (output) rises. On the other hand, implementing this system would imply a change in the head and the tower because they are not adequate to support it. Furthermore, this system is not usually used in lines of small size.

#### 4.3.1.1. Head

The head is the piece located at the end of the screw which supports the die. Its internal profile must ensure a fluent flow to the die. The design of this part is very important; it is designed so that the polymer flows at a continuous speed. The following figure shows a scheme of one head.



**Figure 4.5.** Scheme of a three-layer head.

- |                    |               |                        |
|--------------------|---------------|------------------------|
| 1- Sump.           | 3- Die.       | 5- Die centering bolt. |
| 2- Retaining ring. | 4- Vent hole. | 6- Breaker plate.      |

The melted polymer is molded when it passes through the die. There are many types of nozzles. In this process a cylindrical one is used. Figure 4.5 shows the three main parts: the inlet channel, the distributor and the outlet area.

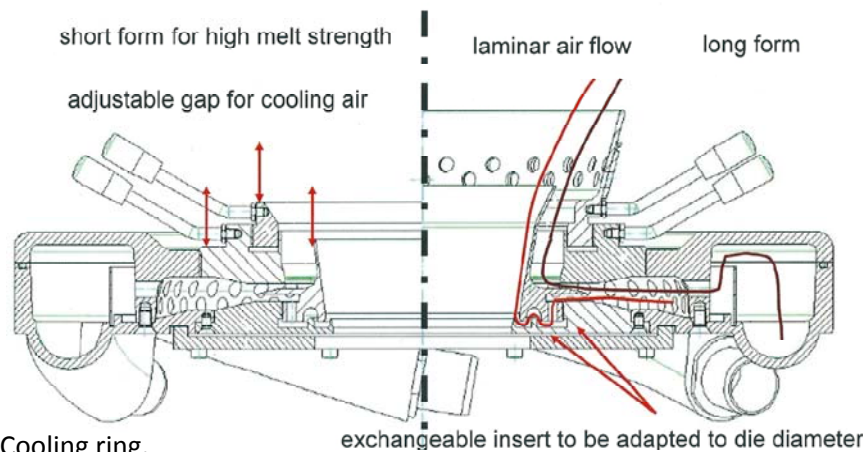
#### 4.3.2. Cooling ring

The cooling ring is one of the key pieces in the coextrusion line. Its function is to refrigerate the bubble providing cold air in the external side of the bubble. Therefore, it is responsible for external cooling of the bubble.

The used coextrusion line has a cooling ring; the proposal is to switch it to a dual lip air ring with air guiding inserts for a more laminar air flow along the bubble. This system allows to drive the cold air better because the ring has an adjustable gap for the cooling air. Moreover, an exchangeable insert can be adapted to the die diameter obtaining a laminar air flow.

All these improvements in the ring are aim to increase the cooling effectiveness. Driving the air with more precision helps to stabilize the bubble more easily and faster. However, this change will allow to increase the output by a small percentage, 5 or 10% at most, but the advantage is that it does not require to change other parts of the coextrusion line; it only requires to replace the cooling ring. And another complementary advantage is that the film

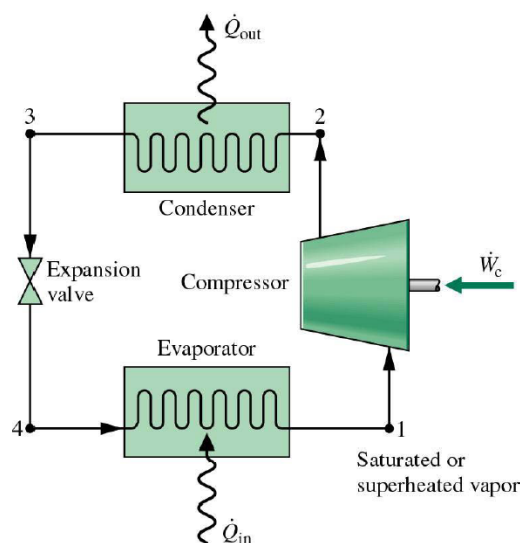
properties such as transparency, gloss and strength improve slightly with this kind of ring due to the higher stabilization of the film bubble. A scheme is shown in figure 4.6.



**Figure 4.6.** Cooling ring.

### 4.3.3. Refrigeration cycle

In order to cool the air used in the air ring or in the IBC system, the extrusion line includes an extra system which consists of a compression refrigeration cycle which uses R134a as a refrigeration gas so as to cool water. The scheme of the process is the following:



**Figure 4.7.** Compression refrigeration cycle.

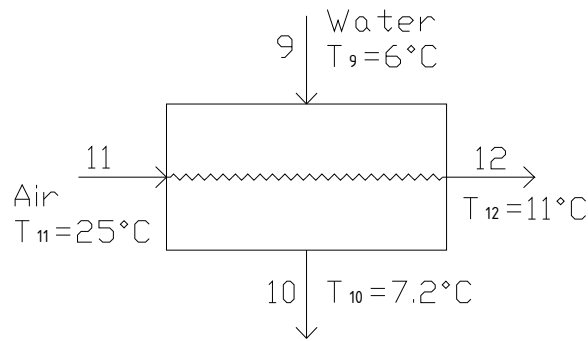
The compressor pushes the gas against the condenser where the gas gives out heat to the atmospheric air which is fed using a ventilator. This heat exchange implies that the gas changes from gas to liquid. The condenser air is fed at 25°C and leaves at 40°C.

Outside the condenser we have under-cooled liquid. Now, the fluid is pushed to the expansion valve and is injected to the evaporator.

This is an isenthalpic valve and the fluid is expanded changing from liquid to gas due to the drop pressure. Then, in the evaporator, the fluid receives heat from the water which is

supplied at 15°C so as to carry out the heat exchange. Then, the fluid is heated and changes to saturated vapor. Finally, the compressor aspirates the gas and the process restarts.

The most interesting part, in terms of extrusion, is the evaporator which refrigerates water from 15 to 6°C. This outlet stream goes to one heat exchanger which has another stream of atmospheric air. At this point, the cold water refrigerates the air which goes to the air ring and/or to the IBC system. The following scheme shows the mentioned exchanger:



**Figure 4.8.** Water-Air heat exchanger scheme.

The following table contains the flows and the exchanged heat of the refrigeration cycle combined with the air- water heat exchanger.

**Table 4.1.** Process flows.

Streams	Fluid	$\dot{m}$ (kg/s)	Q (kW)
2-3	R134a	0.10	-24.7
4-1	R134a	0.10	24.2
6-7	Air	1.64	24.7
8-9	Water	0.65	-24.2
9-10	Water	0.26	3.09
11-12	Air	0.22	-3.09

The compressor develops a power of 0.52kW and the valve 0kW (adiabatic valve).

And finally, table 4.2 shows the conditions of the refrigeration cycle streams<sup>3</sup>.

**Table 4.2.** Parameters of the compression refrigeration cycle.

Stream	P (bar)	T (°C)	x	h (kJ/kg)	s (kJ/kg·K)	v (m <sup>3</sup> /kg)
1	22	71.8	1	281	0.90	8.24·10 <sup>-4</sup>
2	27	84.1	100	286	0.90	6.54·10 <sup>-3</sup>
3	27	-10.0	-100	39.1	0.15	7.49·10 <sup>-4</sup>
4	22	-9.89	-100	39.1	0.15	7.50·10 <sup>-4</sup>

<sup>3</sup> The parameters have been calculated simulating the refrigeration cycle with EES program. See annex A.1.2.

#### 4.3.4. Analysis of the alternatives

We compare the different proposed alternatives to improve the refrigeration process.

**Table 4.3.** Priority matrix of the improvement alternatives.

PARAMETERS	ALTERNATIVES		
	PRE-COOL AIR	NEW COOLING RING	ADD IBC SYSTEM
Equipment	Heat exchanger and gas refrigeration cycle.	Dual lip air ring with air guiding inserts.	Internal Bubble Cooling device with concentric pipes.
Refrigeration improvement	5-10%	5-10%	≈50%
Cost	€€	€€	€€€€
Availability	Good	Good	Poor for this size
Space	Plenty of space (Outdoor space to install the cycle equipments is required)	No space (Consists of replacing the ring)	Little space (Implementation in the machine as such)
Pressure	Low-Medium pressure (27 bar in refrigeration cycle)	Low (atmospheric)	Low (atmospheric)
Extension	High	Low (Add some devices)	Low (Add more sensors)
Maintenance	High	Low	Low

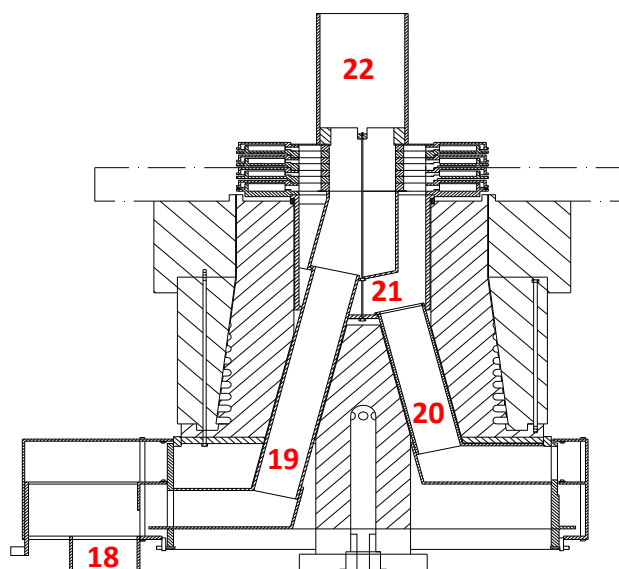
Analyzing the different alternatives, we observe that all of them are a good option. In terms of effectiveness it is clear. The only option which offers the possibility to solve the current problem of the coextrusion line is the implementation of an IBC system. An improvement of 50% is significant. The other improvements help to get a higher-quality film more than the refrigeration because an improvement of maximum a 10% is not appreciable in terms of refrigeration. However, referring to film quality it is, because the bubble is more stable when presents fewer instabilities. Therefore, the conclusion is to implement both first systems to improve the film quality and refrigeration in a small degree.

Considering the price, implementing an IBC system is an important investment which requires to an in-depth study. Moreover, the size of this line is an additional difficulty to purchase the system because the supplier is still studying the possibility to develop this system for small lines. In case of purchase, we will need to buy and adapt a new tower to support the ring, the IBC system and the basket and nip-rolls.

In conclusion, the best option is to install a heat exchanger with a gas refrigeration cycle to cool the air and replace the cooling ring with another one which can drive the air flow better. Finally, if we really want to increase more the output to reproduce still better industrial conditions in the fabrication laboratory, attention should be paid to the market so as to implement an IBC system when the supplier decide unto work in this direction.

#### 4.3.5. Sizing pipes and accessories

The following figure shows the lateral section of the IBC system. The material used is steel because the fluid which must pass through the tubes is air and it does not present any contraindication in contact with steel, such as could be corrosion or flammable problems. The cold air is introduced through pipes 20 and 21. The hot air leaves through 22, 19 and 18.



**Figure 4.3.** Pipes' sectional view of IBC system.

In order to design pipes for the IBC has been taken into account the following hypotheses:

- The maximum air speed is about 40 m/s.
- The flow which passes through pipe 22 must be 3 times the flow in pipes 19 or 20.
- Pipe 21 must be wide enough to contain pipe 22 and the devices through which the air get into the bubble.
- Pipe 18 should be a bit wider than pipes 19 and 20 so as to ensure the correctly extraction of hot air.

Once the mentioned premises have been considered, the pipes are sized and standardized.

Tables 4.1 and 4.2 show the results.

**Table 4.1.** Sizing pipes.

PIPE	Flow (m <sup>3</sup> /h)	speed (m/s)	Area (m <sup>2</sup> )	Internal diameter (mm)
19 (outlet air)	90	40	8100	101.6
20 (inlet air)	90	40	8100	101.6
22 (outlet air)	270	40	24300	175.9
21 (central IBC)	607.5	40	54675	263.8
18(inlet air, down-left)	152.1	40	13689	132.0

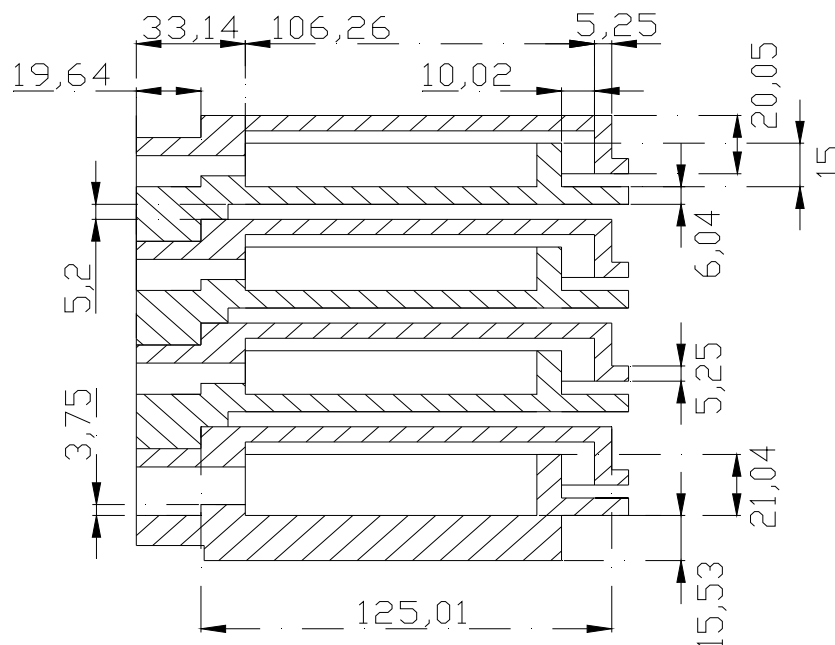
**Table 4.2.** Standardization pipes.

PIPE	Internal diameter (mm)	Thickness (mm)	Area (m <sup>2</sup> )	Flow (m <sup>3</sup> /h)
19 (outlet air)	102.3	6.020	102.3	91.30
20 (inlet air)	102.3	6.020	102.3	91.30
22 (outlet air)	202.7	8.180	202.7	358.6
21 (central IBC)	303.2	10.31	303.2	802.2
18(inlet air, down-left)	154.1	7.110	154.1	207.2

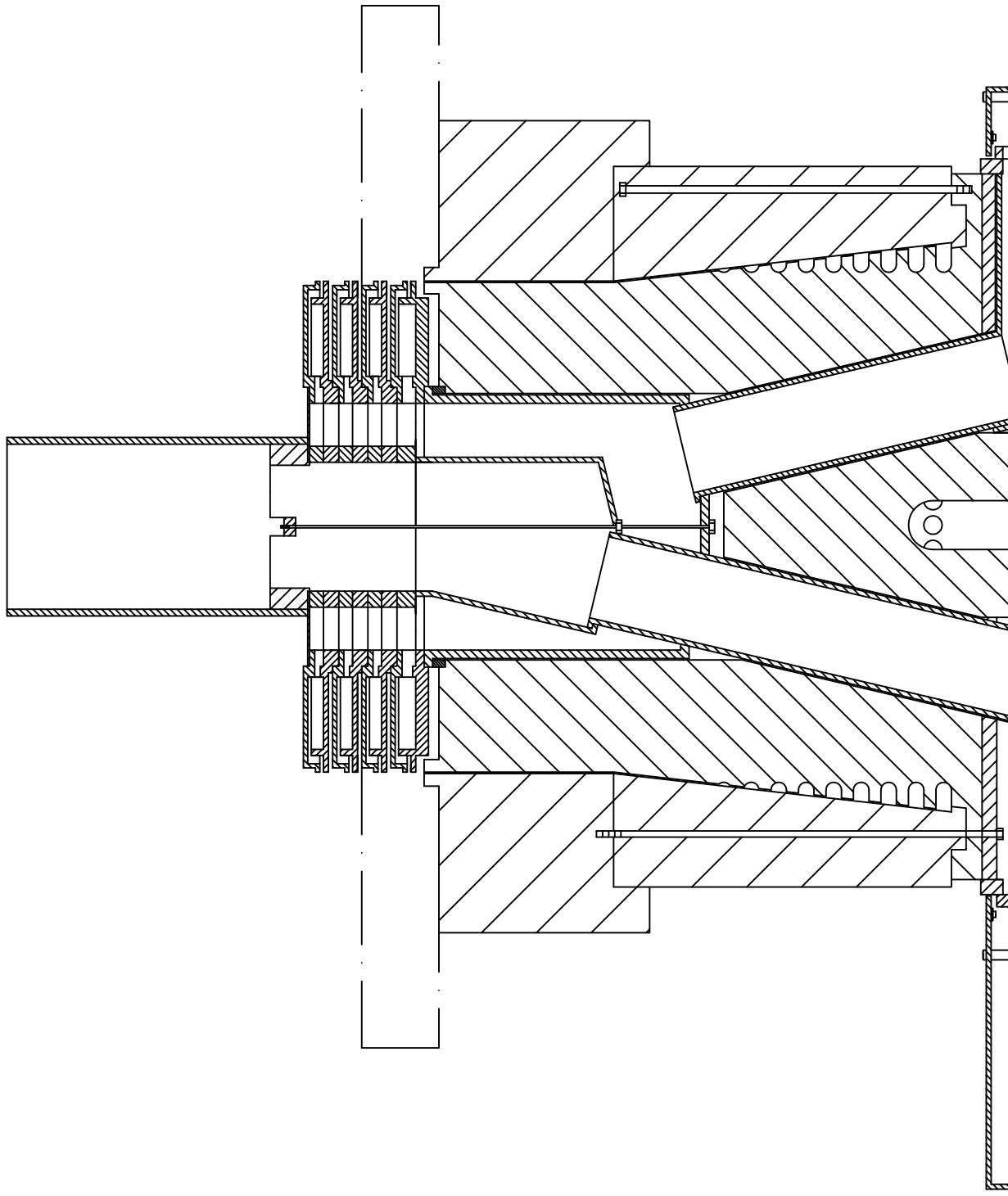
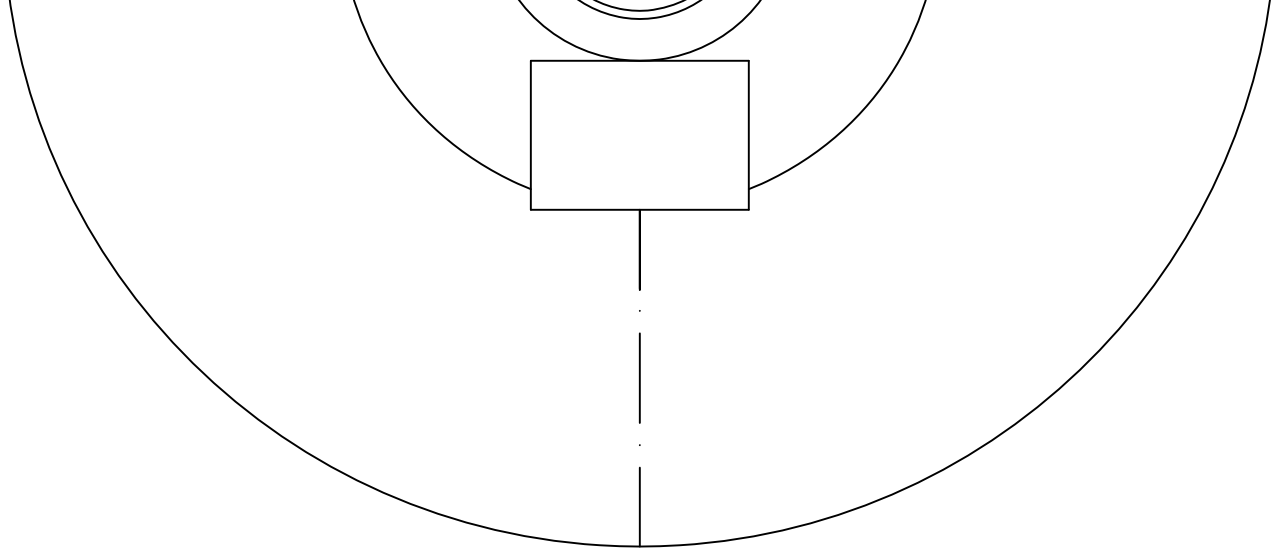
These pipes do not require any insulation material because the operation temperature ranges from 2 - 5 °C to 20 - 30 °C and there are no risks working at these temperatures.

Finally, next to the outlet central pipe of the IBC system there is a place where air inlet devices are located. These devices are structures through which the air passes and it is driven along the inferior part of the bubble with the aim to refrigerate it faster. Then the air goes to the superior part continuing this process.

Figure 4.4 shows four of these devices one over the other ensuring good internal refrigeration. The first structure is a bit different because it needs to be adapted to the tube in order to form a closed area whereas the other three are identical. The material used is the same, steel, because the fluid is also air.

**Figure 4.4.** Air inlet devices (dimensions expressed in mm).

It is important consider that the IBC measures must be modify depending on the size of the die used in the coextrusion line.





All at once, a refilling system is used to introduce the material into the hoppers. This requires one tube per hopper so as to feed the extruders. In this case the hypotheses which have been considered are the following:

- There are different samples and the combinations which present higher and lower specific gravity have been selected. Then, the parameters are calculated.
- A speed of 0.5 m/s has been chosen.
- The material used is plastic because the materials which must pass through the tubes are solid polymers at environmental conditions and a flexible tube seems the best option.

The results can be appreciated in the following tables.

**Table 4.3.** Sizing pipes.

Mixtures	$\rho_{\text{mixutre}}$ (kg/m <sup>3</sup> )	Speed (m/s)	Flow (kg/s)	Flow (m <sup>3</sup> /s)	Area (m <sup>2</sup> )	Internal diameter (mm)
LLDPE/LDPE (80:20)	918.6	0.5	0.3	$3.27 \cdot 10^{-4}$	$6.54 \cdot 10^{-4}$	28.86
LLDPE/SBS 2/CaCO <sub>3</sub> (65:15:20)	1289	0.5	0.3	$2.94 \cdot 10^{-4}$	$5.88 \cdot 10^{-4}$	27.37

Once the required diameter has been calculated, the next step is to search tubes in catalogues<sup>4</sup> to standardize the diameter. Table 4.4 shows the final results.

**Table 4.4.** Standardization pipes.

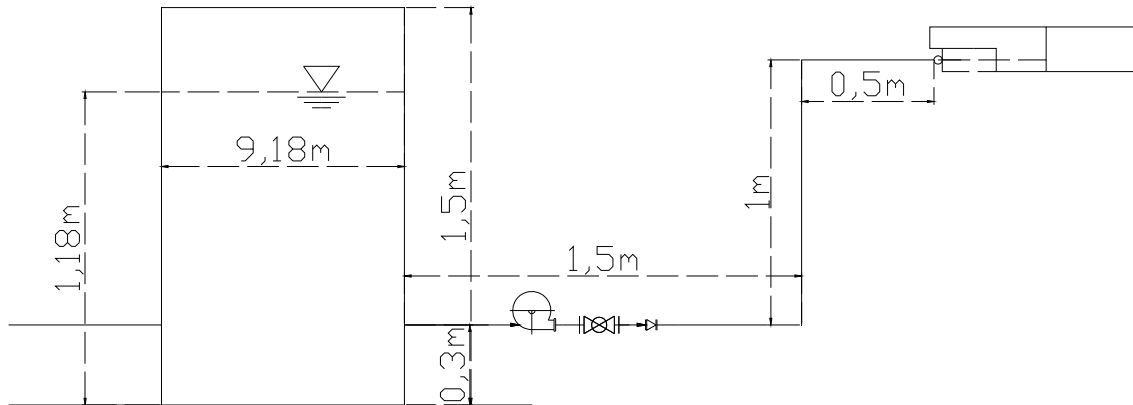
Nominal diameter (mm)	Internal diameter (mm)	Thickness (mm)	Area (m <sup>2</sup> )
40.0	31.2	8.80	$7.65 \cdot 10^{-4}$

#### 4.3.6. Pumps design

In order to feed water to the refrigeration system of the extruders we need to install one pump per extruder. In the outside, one tank stores the water which is used in the plant. at atmospheric pressure. The water is pressurized when it leaves to the tank at 6 kg/cm<sup>2</sup> because, although the desirable pressure to work is a 4 kg/cm<sup>2</sup>, the water suffers a drop pressure due to the different element through which it passes during the transport.

The first step is to identify the system, then design the pipes and finally determine if any pumps are required or not. If it is necessary, the last step is to design the pump. Figure 4.5 shows the studied system.

<sup>4</sup> The catalogue used is by CAPSA. Link: [http://www.capsa.com.ni/pdf/SISTEMAS\\_TUBERIA\\_ELECTRICA.pdf](http://www.capsa.com.ni/pdf/SISTEMAS_TUBERIA_ELECTRICA.pdf)



**Figure 4.5.** System.

**Table 4.5.** Fluid properties and basic parameters.

Extruder	Material	$\rho$ (kg/m <sup>3</sup> )	$C_p$ (kJ/kg·°C)	Viscosity (Pa·s)	Flow (kg/h)	$T_1$ (°C)	$T_2$ (°C)	$\Delta T$ (°C)
1 and 3	Resin	1289	23.5	-	1.6	300	50	-250
2	Resin	1289	23.5	-	7.7	300	50	-250
1 and 3	Water	998	4.184	$1.02 \cdot 10^{-3}$	224	20	30	10
2	Water	998	4.184	$1.02 \cdot 10^{-3}$	1081	20	30	10

Once the heat exchange has been determined by doing a basic energy balance, we proceed to calculate the pipes diameter and to standardize<sup>5</sup> them.

**Table 4.6.** Design parameters.

Extruder	Q (kJ/h)	Water flow (m <sup>3</sup> /h)	v (m/s)	A (m <sup>2</sup> )	$D_{int}$ (mm)	$D_{stand.}$ (mm)	$A_{stand.}$ (m <sup>2</sup> )	Thickness (mm)
1 and 3	9400	0.08						
2	45237	0.33	1.0	$1.0 \cdot 10^{-4}$	11.3	15.8	$2.0 \cdot 10^{-4}$	2.77

Then, we associate a drop pressure with each element and determine the total drop pressure ( $h_L$ ) with Darcy equation.

$$h_L = \left( f \cdot \frac{L}{D} + \sum K \right) \cdot \frac{v^2}{2g} \quad (4.1)$$

Where K is  $f_T$  (friction coefficient) multiplied by  $L_e/D$  (equivalent length) of each element. V is the average speed, f the friction factor of the pipe extracted from the Moody diagram<sup>6</sup> and L/D the length and diameter of the pipe.

<sup>5</sup> <http://www.pumpgroup.com/productdisp1.php?companyid=12&categoryid=71&pid=229>

**Table 4.7.** Drop pressure element and design parameters.

L (m)	Rugosity (mm)	$L_e/D$ elbow 90°	$L_e/D$ valve	$L_e/D$ check valve	$f_T$	$h_L$ (m)
73	0.0024	30	150	135	0.032	5.08
Re	$h_{\text{tank}}$ (m)	$K_{\text{elbow 90°}}$	$K_{\text{valve}}$	$K_{\text{check valve}}$	$K_{\text{tube input}}$	$K_{\text{tube output}}$
$1.55 \cdot 10^4$	1.5	0.75	3.75	3.4	0.5	1.0

Finally, by writing the mechanical balance we determine the pump height ( $h_{\text{PUMP}}$ ) and its power using Grundfos® catalogues<sup>7</sup>.

$$\frac{P_1}{\rho_1 g} + \frac{v_1^2}{2g} + z_1 + h_{\text{PUMP}} - h_L = \frac{P_2}{\rho_2 g} + \frac{v_2^2}{2g} + z_2 \quad (4.2)$$

The following hypotheses have been considered:

- The water is static at the top of the tank ( $v_1=0$ ).
- The z reference is the water level tank ( $z_1=0$ ).
- The tank is not pressurized ( $P_1=\text{atmospheric pressure}$ ).

**Table 4.8.** Pump parameters.

$P_1$ (Pa)	$P_2$ (Pa)	$v_1$ (m/s)	$v_2$ (m/s)	$z_1$ (m)	$z_2$ (m)	$h_{\text{PUMP}}$ (m)	$P_{\text{PUMP}}$ (W)	$\eta$ (%)	$P_{\text{REAL}}$ (W)
$10^5$	$4 \cdot 10^5$	0	1.0	0	0.5	52	51.4	35	150

The selected pump is a CR1s with a flow range of 0.3 - 1.1 m<sup>3</sup>/h. It is made of stainless steel AISI 316 and it is connected with the pipes through an oval flange DN25/DN32. The corresponding specification sheet is shown in annex A.1.4.

#### 4.3.7. Instrumental design and control

##### 4.3.7.1. Control variables of the process

The following table includes the process variables which must be controlled, with the set-point and the operation range.

**Table 4.9.** Control variables.

Controlled variables	Optimal Set-point	Operation Range	Units
Level of the extruders	0.75	0.70-0.80	m
Water flow of the extruders	*	*	m <sup>3</sup> /h
Temperature in ZONE-1 of the extruders	180	178-182	K
Temperature in ZONE-2 of the extruders	220	218-222	K
Temperature in ZONE-3 of the extruders	230	228-232	K
Temperature in ZONE-4 of the extruders	235	233-237	K

<sup>6</sup> <http://people.msoe.edu/~tritt/be382/graphics/Moody.png>

<sup>7</sup> <http://es.grundfos.com/products/find-product/cr.html#catálogos>

**Table 4.9. Control variables. (Continuation)**

Controlled variables	Optimal Set-point	Operation Range	Units
Temperature in the filter of the extruders	235	233-237	K
Temperature in the adapter of the extruders	235	233-237	K
Temperature in die of the extruders	240	238-242	K
Pressure in ZONE-1 of the extruders	1 (1)**	1-5 (1-5)**	bar
Pressure in ZONE-2 of the extruders	20 (60)**	15-25 (40-80)**	bar
Pressure in ZONE-3 of the extruders	70 (110)**	65-75 (90-130)**	bar
Pressure in ZONE-4 of the extruders	120 (180)**	115-125 (160-200)**	bar
Pressure in the filter of the extruders	145 (240)**	140-150 (220-260)**	bar
Pressure in the adapter of the extruder s	155 (275)**	150-165 (255-295)**	bar
Pressure in die of the extruders	155 (275)**	150-165 (255-295)**	bar
Screw speed of the extruders 1 and 3	35	32-38	rpm
Screw speed of the extruder 2	85	82-88	rpm
Air flow of the cooling ring	2000	1950-2050	m <sup>3</sup> /h
Air flow of the IBC system	500	475-525	m <sup>3</sup> /h
Temperature right-inferior area of the bubble	15	10-20	K
Temperature left-inferior area of the bubble	15	10-20	K
Bubble diameter	0.25	$\pm 5 \cdot 10^{-3}$	m

\*Depending on the mixture.

These are the variables of the process which require to be controlled to ensure the manufacture of a good quality film satisfying the safety conditions. If the temperature or the pressure of the extrusion process rises uncontrollably important damages can arise because the equipment is working at high temperatures and pressures. In the following sections, the control loops, required by the process, are explained in detail.

#### **4.3.7.2. Control strategy characterization**

##### **4.3.7.2.1. Hopper**

Controlling the feed level of the material is essential in order to work in continuous. The presence of pellets in the hopper is indispensable, if not the extruder can get damaged. The hopper includes a laser sensor (radar) which determines the filling percentage by means of the distance difference. Each hopper includes one: LT-01, LT-02 and LT-03.

The sensors send the measures to their controllers (LC-01, LC-02 and LC-03) which compare the values with the set-point and, if it is necessary, react by turning on the aspirators so as to refill the hoppers. It all depends on the filling percentage. Using a computer, we fix a minimum and a maximum filling percentage, which appear in table 4.5. When the level is below the minimum, the level controller (LC) gives the order of turning on the aspirator which refills the hopper. If there is no material in the sacks, in which the tubes are, the LC activates on one alarm to alert the operator. This is a simple control loop with reverse

action; when the level decreases below the set-point, the controller reacts increasing the supplied tension of the aspirator so as to turn it on and refill the hopper.

#### **4.3.7.2.2. Extrusion process**<sup>8</sup>

Four temperature (TT-01, TT-02, TT-03 and TT-04) and four pressure sensors (PT-01, PT-02, PT-03 and PT-04) are installed all through the barrel, which contains the screw, in order to control these variables during the extrusion process.

Controlling the temperature is very important because the temperature profile is one essential condition in this process. Depending on the material, this profile must be higher or lower because every material has its own melt temperature which must be achieved so as to guarantee a good flow. The extruders are divided into 4 regions and have electrical resistance packs, small fans (FN-01, FN-02, FN-03 and FN-04) and one auxiliary water refrigeration cycle so as to increase or reduce the temperature in the different areas. This system is used to refrigerate the entire extruder; it is used in the stop procedure. All the temperature sensors are connected with one high selector (HS-04) which takes the highest temperature so as to refrigerate the extruder if it exceeds the set-point. This system prevents that the temperature increases non-stop avoiding dangerous problems. However, the normal use is to refrigerate faster during the stop procedure. In addition, the extruders have three more temperature sensors: one in the filter (TT-13), another in the adapter (TT-14) and another one in the die (TT-15).

The sensors measure the temperature and send a signal to their controller (TC-04, TC-13 and TC-14) connected in cascade with the flow controller (FC-01) of the refrigeration water valve (VLV-01). Except the die temperature sensor which is only connected with FN-06. And TC-13 is also connected with FN-05. This is a direct action control loop; the more temperature, the more opening % of the valve. The control loop with FIT-01 plus FC-01 is a secondary one.

In addition, if the measure is below the set-point, the controllers send the order of activating the corresponding fan which acts as a manipulated variable. Otherwise, if the temperature is too low, the controller will increase the tension supplied to the electric resistances. This is a reverse action loop because the lower the temperature is, the more tension we need to supply to the electric resistances.

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<sup>8</sup> All the references are for extruder 1.

Normally, in order to refrigerate the used systems only fans will be needed, but as table 4.5 shows when facing a bigger difference, the water refrigeration cycle will be switched on opening the valve VLV-01. This control requires an advanced control because this is a secondary control loop connected in cascade with the primary loop which is composed by all the temperature sensors which all end in one HS.

In case of pressure, there is the same number of sensors in the same areas, but in contrast to temperature, where all the sensors are independent and each one has its controller (HS-01), the aim of pressure control is not to elaborate a pressure profile. Here, the most important thing is not to exceed the 350 bar. The entire PT sends its signals to one high selector (HS) which chooses the highest value. This is connected in cascade with the VC-01 of the screw motor and if the pressure exceeds the set-point the VC-01 reacts reducing the feed tension and as a result the screw rpm which are controlled with another loop (VT-01 and VC-01). This is a reverse control loop; when the pressure increases too much, we reduce the supplied tension to the screw motor so as to decrease the rpm. This is an advanced control, too. There is one secondary loop, the VT-01 plus VC-01, which is connected in cascade with one HS where all the pressure measures (primary control loops) end.

All these sensors and controllers are used in every extruder and are listed in table 4.5 with their corresponding set-point and operation interval.

#### **4.3.7.2.3. Cooling ring**

The cooling ring has two inlets of air. In each inlet, there is one FT after the stream valve (FIT-04 and FIT-05). They are connected with their controllers (FC-04 and FC-05). The flow is the measured variable and the opening of the valves (VLV-04 and VLV-05) is the manipulated variable to which the controllers send the order of increasing or decreasing the value depending on the fixed set-point. In addition, the air flow to swell the bubble only includes a FIT (FIT-06). This kind of control is composed by simple control loops, all of them with direct action. When the temperature is too high, the % of valve opening increases so as to introduce more air and to decrease the temperature of the polymeric mass.

#### **4.3.7.2.4. IBC system**

This system includes the same control loop to control the flow (FIT-07 and FC-06), as it is used to control the air flow of the cooling ring. Moreover, it has another loop. There is another variable to consider so as to ensure a good control and to extrude a quality film.

This device incorporates two temperature sensors (TT-22 and TT-23) in order to control the temperature inside the bubble. These measures are sent to one HS-07 which acts over the manipulated variable, which is the opening of the air flow valve VLV-06 with FC-06.

Finally, the IBC system includes sensors to control the variations in tube circumference (SF-01 and SF-02). They determine the error and sending a signal to the valve VLV-06 in order to introduce more or less air into the bubble. The process consists of send a light beam to the bubble walls and measuring the amount of light that reaches the detector.

These loops are the primary control loops which are connected in cascade with the own air flow control of the stream, constituting the secondary loop. The primary one is a direct action loop and the secondary one a reverse action loop.

#### **4.3.7.2.5. Refrigeration cycle**

The scope of the project does not include the control of the refrigeration cycle. However, the air-water heat exchange is. This equipment requires a good control of the temperature so as to ensure a proper heat exchange.

One valve in the water inlet and one temperature sensor in every stream are the basic instrumentation. Then, there are flow meters in the inputs. The flow in the inputs is controlled with a simple loop which acts as a secondary loop. The temperature sensors of the outputs have their temperature controller and if the air temperature is not cold enough, the controller orders to increase the water flow. This control loop is the primary one and it is connected in cascade with the mentioned secondary loop.

#### **4.3.7.2.6. Safety control loops**

If there is a critic process change which implies that the operation parameters are out of the established range, temporarily or permanently, it is required to press the emergency button and switch off the extruders following the emergency plan (see section 6.4).

The set-points limitation of the critic variables is shown in table 4.7.

**Table 4.10.** Critical set-point values.

PARAMETER	CRITICAL VALUE
Temperature	The maximum set-point at the extruder adaptors is 573 K.
Pressure	The maximum pressure at the extruder adaptors is 350 bar.
Screw speed	The maximum set-point is 120 rpm.
Line speed	The maximum set-point speed is 30 m/min.
Amps	The maximum extrusion amperage in extruder 2 (30 mm) is 7 A.
Amps	The maximum extrusion amperage in extruder 3 (25 mm) is 3 A.

The loops to control these critical variables appear in table 4.7. Their controllers always require fixing the set-point below those critical values and display the fixed values to verify that they are always keep far from the critical ones.

#### **4.3.7.3. Instrumentation and control design**

The extrusion line does not require much space, a section of 20 or 25 m<sup>2</sup> is enough, so that, it is easy to send all the signals to only one master computer. At the same time, the line has three panels where the temperature profile and pressure measures can be appreciated to ensure a reliability control.

Moreover, in the PCD the temperature and pressure controllers are drawn separately, but all the inputs and outputs go to the same point where one process controller<sup>9</sup> receives and sends the corresponding signals. This model is designed for 8 zones and has 2 USB ports to send the signal to the computer easily. Then, we can modify the set-points of all the temperatures and pressures along the extruder in a simple way. Each individual extruder is equipped with one of these process controllers. All the control units are connected to the computer which manages all set values and formulation parameters, activates the extruders and records all measured values. The control system also comprises extensive monitoring, diagnostics and alarm utilities.

#### **4.3.7.4. Relation among the controllers**

In this section a summary table is presented in order to understand better the relation between controllers and their parameters.

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<sup>9</sup> <http://www.dynisco.com/extrusion-machine-controllers>

Table 4.11. Control loops.

Name	Controlled variable	Manipulated variable	Controller	Loop	Action	Fail	Set-point	Control
LT-01*	Level of pellets	Tension supplied to the aspirator A-01	LC-01	Primary	Reverse	Open	0.75 m	P
FIT-01*	Water flow F7	Opening of valve VLV-01	FC-01	Secondary	Reverse	Open	** m <sup>3</sup> /h	PI
VT-01	Screw rpm of extruder 1	Tension supplied to motor MR-01	VC-01	Secondary	Reverse	Close	35 rpm	P
VT-02	Screw rpm of extruder 2	Tension supplied to motor MR-02	VC-02	Secondary	Reverse	Close	85 rpm	P
VT-03	Screw rpm of extruder 3	Tension supplied to motor MR-03	VC-03	Secondary	Reverse	Close	35 rpm	P
TT-01*	Extruder temperature at ZONE 1	Tension supplied to fan FN-01	TC-01	Primary	Direct	Open	453 K	PID
		Opening of valve VLV-01	FC-01	Primary	Direct	Open	493 K	PID
TT-02*	Extruder temperature at ZONE 2	Tension supplied to fan FN-02	TC-02	Primary	Direct	Open	453 K	PID
		Opening of valve VLV-01	FC-01	Primary	Direct	Open	453 K	PID
TT-03*	Extruder temperature at ZONE 3	Tension supplied to fan FN-03	TC-03	Primary	Direct	Open	493 K	PID
		Opening of valve VLV-01	FC-01	Primary	Direct	Open	493 K	PID
TT-04*	Extruder temperature at ZONE 4	Tension supplied to fan FN-04	TC-04	Primary	Direct	Open	503 K	PID
		Opening of valve VLV-01	FC-01	Primary	Direct	Open	503 K	PID
PT-01*	Extruder pressure at ZONE 1	Tension supplied to motor MR-01	VC-01	Primary	Reverse	Close	155 bar	PID
PT-02*	Extruder pressure at ZONE 2	Tension supplied to motor MR-01	VC-01	Primary	Reverse	Close	155 bar	PID
PT-03*	Extruder pressure at ZONE 3	Tension supplied to motor MR-01	VC-01	Primary	Reverse	Close	155 bar	PID
PT-04*	Extruder pressure at ZONE 4	Tension supplied to motor MR-01	VC-01	Primary	Reverse	Close	155 bar	PID
TT-13*	Temperature at the filter	Tension supplied to fan FN-04	TC-04	Primary	Direct	Open	508 K	PID
		Opening of valve VLV-01	FC-01	Primary	Direct	Open	508 K	PID
TT-14*	Temperature at the adapter	Tension supplied to fan FN-05	TC-13	Primary	Direct	Open	508 K	PID
		Opening of valve VLV-01	FC-01	Primary	Direct	Open	508 K	PID
TT-15*	Temperature at the die	Tension supplied to fan FN-06	TC-14	Primary	Direct	Open	508 K	PID
PT-13*	Pressure at the die	Tension supplied to motor MR-01	VC-01	Primary	Reverse	Close	155 bar	PID
FIT-04	Air flow F14	Opening of valve VLV-04	FC-04	Secondary	Reverse	Open	2000 m <sup>3</sup> /h	PI
FIT-05	Air flow F15	Opening of valve VLV-05	FC-05	Secondary	Reverse	Open	2000 m <sup>3</sup> /h	PI
FIT-07	Air flow F20	Opening of valve VLV-07	FC-06	Secondary	Reverse	Open	500 m <sup>3</sup> /h	PI
TT-22	Bubble temperature (at left)	Opening of valve VLV-07	FC-07	Primary	Direct	Open	513 K	PI
TT-23	Bubble temperature (at right)	Opening of valve VLV-07	FC-07	Primary	Direct	Open	513 K	PI
SF-01	Bubble diameter	Opening VLV-06	FC-06	Primary	Direct	Closed	** m	PI
SF-02	Bubble diameter	Opening VLV-06	FC-06	Primary	Direct	Closed	** m	PI

\*This control loop is exactly the same in extruders 2 and 3. The only difference is the ID number; show the PCD to identify properly. \*\*Depending on mixture.

#### 4.3.7.5. Control instruments relation

In order to control the variables mentioned in table 4.5 is necessary use some different measurement instruments and the corresponding controllers. All of them are recollected in the following table with the used valves.

**Table 4.12.** Instrumentation list.

TAG	SERVICE	TYPE	FABRICANT	MODLE
LT-01	Level sensor	Radar	SICK	DT20-P130B1000
LT-02	Level sensor	Radar	SICK	DT20-P130B1000
LT-03	Level sensor	Radar	SICK	DT20-P130B1000
LC-01	Level controller	-	WEKA	EM-15
LC-02	Level controller	-	WEKA	EM-15
LC-03	Level controller	-	WEKA	EM-15
AL-01	Alarm buzzer	Audible	SWITCHES PLUS	55-04301
AL-02	Alarm buzzer	Audible	SWITCHES PLUS	55-04301
AL-03	Alarm buzzer	Audible	SWITCHES PLUS	55-04301
FIT-01	Flow indicator sensor	Ultrasounds	SICK	FFUS15-1G1/O
FIT-02	Flow indicator sensor	Ultrasounds	SICK	FFUS15-1G1/O
FIT-03	Flow indicator sensor	Ultrasounds	SICK	FFUS15-1G1/O
FC-01	Flow controller	-	MKS	MKS-1579
FC-02	Flow controller	-	MKS	MKS-1579
FC-03	Flow controller	-	MKS	MKS-1579
VLV-01	Ball valve	High pressure valve	TECVAL	VB-12
VLV-02	Ball valve	High pressure valve	TECVAL	VB-12
VLV-03	Ball valve	High pressure valve	TECVAL	VB-12
TT-01	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-02	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-03	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-04	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-05	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-06	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-07	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-08	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-09	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-10	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-11	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-12	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TC-01	Temperature controller	-	DYNISCO	71-1243-210
TC-02	Temperature controller	-	DYNISCO	71-1243-210
TC-03	Temperature controller	-	DYNISCO	71-1243-210
TC-04	Temperature controller	-	DYNISCO	71-1243-210
TC-05	Temperature controller	-	DYNISCO	71-1243-210
TC-06	Temperature controller	-	DYNISCO	71-1243-210
TC-07	Temperature controller	-	DYNISCO	71-1243-210
TC-08	Temperature controller	-	DYNISCO	71-1243-210
TC-09	Temperature controller	-	DYNISCO	71-1243-210
TC-10	Temperature controller	-	DYNISCO	71-1243-210

**Table 4.12.** Instrumentation list. (continuation)

TAG	SERVICE	TYPE	FABRICANT	MODLE
TC-11	Temperature controller	-	DYNISCO	71-1243-210
TC-12	Temperature controller	-	DYNISCO	71-1243-210
HS-04	High selector	-	SIL	112-21A
HS-05	High selector	-	SIL	112-21A
HS-06	High selector	-	SIL	112-21A
PT-01	Pressure transducer	Differential	HONEYWELL	FP-2000
PT-02	Pressure transducer	Differential	HONEYWELL	FP-2000
PT-03	Pressure transducer	Differential	HONEYWELL	FP-2000
PT-04	Pressure transducer	Differential	HONEYWELL	FP-2000
PT-05	Pressure transducer	Differential	HONEYWELL	FP-2000
PT-06	Pressure transducer	Differential	HONEYWELL	FP-2000
PT-07	Pressure transducer	Differential	HONEYWELL	FP-2000
PT-08	Pressure transducer	Differential	HONEYWELL	FP-2000
PT-09	Pressure transducer	Differential	HONEYWELL	FP-2000
PT-10	Pressure transducer	Differential	HONEYWELL	FP-2000
PT-11	Pressure transducer	Differential	HONEYWELL	FP-2000
PT-12	Pressure transducer	Differential	HONEYWELL	FP-2000
HS-01	High selector	-	SIL	112-21A
HS-02	High selector	-	SIL	112-21A
HS-03	High selector	-	SIL	112-21A
VT-01	Ratemeter	Board	SIMEX	STI-94
VT-02	Ratemeter	Board	SIMEX	STI-94
VT-03	Ratemeter	Board	SIMEX	STI-94
VC-01	Amperage controller	-	DYNISCO	ATC880
VC-02	Amperage controller	-	DYNISCO	ATC880
VC-03	Amperage controller	-	DYNISCO	ATC880
TT-13	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-14	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-15	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-16	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-17	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-18	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-19	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-20	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TT-21	Temperature sensor	Thermocouple	GUEMISSA	Pt100-STSN
TC-13	Temperature controller	-	DYNISCO	71-1243-210
TC-14	Temperature controller	-	DYNISCO	71-1243-210
TC-15	Temperature controller	-	DYNISCO	71-1243-210
TC-16	Temperature controller	-	DYNISCO	71-1243-210
TC-17	Temperature controller	-	DYNISCO	71-1243-210
TC-18	Temperature controller	-	DYNISCO	71-1243-210
TT-22	Temperature sensor	Wireless sensor	BEANAIR	V1R0
TT-23	Temperature sensor	Wireless sensor	BEANAIR	V1R0
SF-01	Distance sensor	Photoelectric	IFM	OGHLFPKG
SF-02	Distance sensor	Photoelectric	IFM	OGHLFPKG
HS-07	High selector	-	SIL	112-21A
PT-13	Pressure transducer	Differential	HONEYWELL	FP-2000

**Table 4.12.** Instrumentation list. (continuation)

TAG	SERVICE	TYPE	FABRICANT	MODLE
PT-14	Pressure transducer	Differential	HONEYWELL	FP-2000
PT-15	Pressure transducer	Differential	HONEYWELL	FP-2000
PC-13	Pressure controller	-	DYNISCO	71-1243-210
PC-14	Pressure controller	-	DYNISCO	71-1243-210
PC-15	Pressure controller	-	DYNISCO	71-1243-210
PI-16	Pressure indicator	Bourdon manometer	MARSH	J0472
PI-17	Pressure indicator	Bourdon manometer	MARSH	J0472
PI-18	Pressure indicator	Bourdon manometer	MARSH	J0472
PI-19	Pressure indicator	Bourdon manometer	MARSH	J0472
PI-20	Pressure indicator	Bourdon manometer	MARSH	J0472
PI-21	Pressure indicator	Bourdon manometer	MARSH	J0472
PI-22	Pressure indicator	Bourdon manometer	MARSH	J0472
PC-13	Pressure controller	-	DYNISCO	71-1243-210
PC-14	Pressure controller	-	DYNISCO	71-1243-210
PC-15	Pressure controller	-	DYNISCO	71-1243-210
PC-13	Pressure controller	-	DYNISCO	71-1243-210
PC-14	Pressure controller	-	DYNISCO	71-1243-210
PC-15	Pressure controller	-	DYNISCO	71-1243-210
PC-16	Pressure controller	-	DYNISCO	71-1243-210
FIT-04	Flow indicator sensor	Ultrasounds	SICK	FFUS15-1G1/O
FIT-05	Flow indicator sensor	Ultrasounds	SICK	FFUS15-1G1/O
FIT-06	Flow indicator sensor	Ultrasounds	SICK	FFUS15-1G1/O
FIT-07	Flow indicator sensor	Ultrasounds	SICK	FFUS15-1G1/O
FC-04	Flow controller	-	MKS	MKS-1579
FC-05	Flow controller	-	MKS	MKS-1579
FC-06	Flow controller	-	MKS	MKS-1579
FC-07	Flow controller	-	MKS	MKS-1579
VLV-04	Ball valve	Miniature valve	TECVAL	VB-12
VLV-05	Ball valve	Miniature valve	TECVAL	VB-08
VLV-06	Ball valve	Miniature valve	TECVAL	VB-08
VLV-07	Ball valve	Miniature valve	TECVAL	VB-08
CHK-01	Check valve	Poppet check valve	TECVAL	VR-01
CHK-02	Check valve	Poppet check valve	TECVAL	VR-01
CHK-03	Check valve	Poppet check valve	TECVAL	VR-01

#### 4.3.7.6. Lever frame system

The blown coextrusion line includes a device in order to avoid dangerous situations. The critical set-point values are shown in table 4.6. If the screw speed increases uncontrollably, the temperature and pressure will increase and the amperage consumption too. Then the extrusion process can exceed those limit values and affect the safety of the installation and the personal. Therefore, apart from the specific controllers, the line includes an electric panel to cut the power supplied to the screw motor and electrical resistances and stop the process. If not the excessive screw revolutions can lead to critical situations.

The system consists of a motor controller which is a combination of a magnetothermic switch<sup>10</sup> with a magnetothermic counter<sup>11</sup>. This switch must have a high interruption capacity. It switches on automatically when the tension overpasses the established limit. Critical pressure is due to excessive screw speed which stops when the switch turns on because no power is supplied to the motor of the extrusion line. This system is connected with all the extruders and if there is a problem with one extruder all of them are stopped.

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<sup>10</sup> [http://www.gepowercontrols.com/es/resources/literature\\_library/catalogs/downloads/InstalCat\\_ES\\_final\\_LR.pdf](http://www.gepowercontrols.com/es/resources/literature_library/catalogs/downloads/InstalCat_ES_final_LR.pdf)

<sup>11</sup> <http://www.carotron.com/dcdrives/elitprogen/>

## 5. PROJECT

This project includes three different studies. The first consist of an analysis of the components one by one separately. One sample per product was sent to Terneuzen laboratories to characterize them in order to know the main properties.

The second is a preliminary study whose purpose is to study the behavior of the SBS molecules, SBS 1 and SBS 2, combined with specific LLDPEs, LLDPE A and LLDPE B. It is very important to study this because it is a non-common polymer in the extrusion processes in the laboratory and the compatibility with all LLDPEs is not well understood.

And the third is the main study, for which the goal is to evaluate in more detail the effect of SBS in a LLDPE matrix type using the results from the second study.

### 5.1. Characterization of raw materials rheology

#### 5.1.1. Description

First of all, it is important to analyze the raw materials. For this purpose, they were sent to the rheology laboratory in Dow Benelux to determine rheological properties and then the experimental results were compared with the theoretical.

#### **Study #1**

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**Title:** LLDPE and SBS pellets rheology.

**Description:** Two LLDPE and two SBS samples to be used in blends study, blown film.

**Purpose:** Characterize raw materials.

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In the study #2, explained in section 5.2, 4 different materials, two LLDPE and two SB Copolymers (SBC), were mainly used.

- LLDPE A
- LLDPE B
- SBS 1
- SBS 2

LLDPE A is a Ziegler-Natta Lineal Low Density Polyethylene (ZN-LLDPE) resin produced using a Ziegler-Natta process which presents a good combination of optical properties, tear strength and sealability. The resin, with a density of 0.917 g/cm<sup>3</sup> and a melt index (MI) of 0.7 g/10 min, also offers a good bubble stability allowing film production at high outputs.

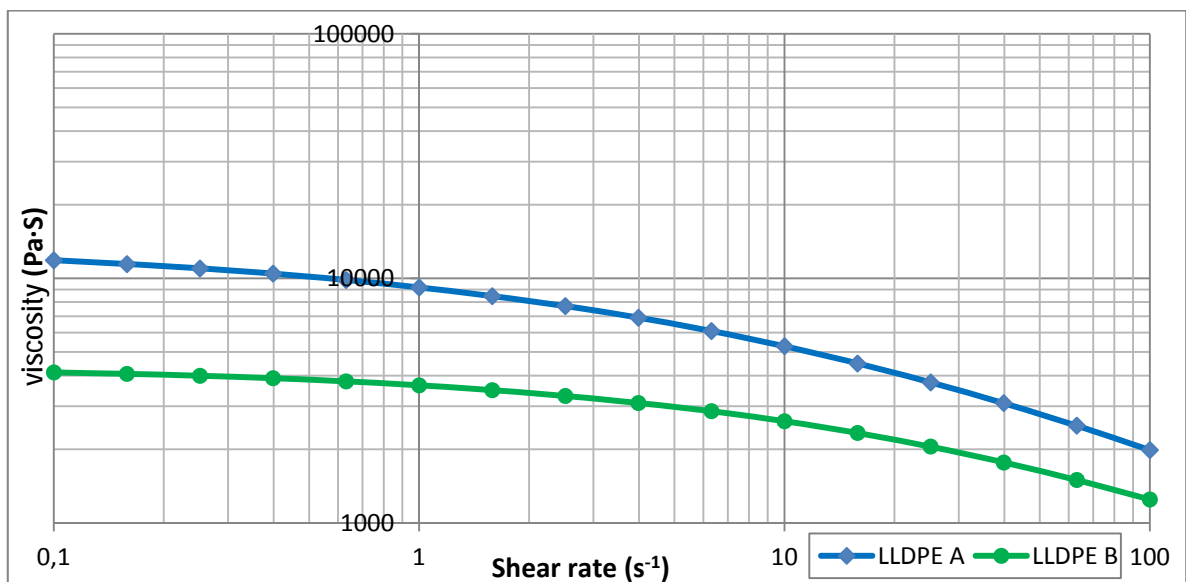
LLDPE B is another ZN-LLDPE resin produced through a ZN process. This resin is an ethylene and 1-octene copolymer and offers an excellent balance of optical and mechanical properties.

SBS 1 and SBS 2, which are Styrene-Butadiene Copolymers (SBC), offer an excellent balance of performance and cost-effectiveness. SBS 1 and SBS 2 present a density of 1.01 and 1.02 g/cm<sup>3</sup> and a MI of 7.5 and 10 g/10 min respectively.

These copolymers bridge the gap between high-cost, clear engineering plastics and low-cost but brittle plastics, such as crystal polystyrene. Moreover, they are easily processed by extrusion or moulding techniques. This kind of resins can be blended with a wide number of polymers to suit performance to specific applications.

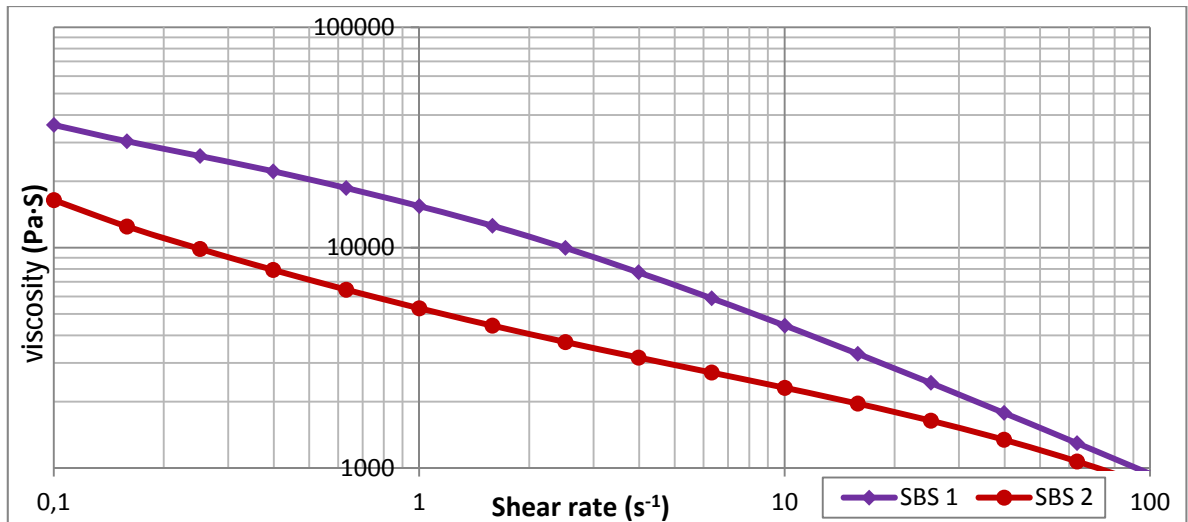
### 5.1.2. Results & discussion

The features which have been evaluated are shown in the following graphs.



**Figure 5.1.** LLDPEs used in study #2.

In figure 5.1, the viscosity differences can be appreciated with regards to shear rate. On the one hand, at lower shear rate LLDPE A viscosity is more than twice the LLDPE B viscosity. This difference is due to the melt index (MFI) of the LLDPEs, viscosity and MFI are reverse proportional and the lower the MFI is, the higher the viscosity is, and viceversa. On the other hand, when the shear rate increases this difference decreases. It is normal because molten polyethylene is not a Newtonian fluid and shows shear thinning. The purpose is to work with two LLDPEs with different specification in order to compare the results and determine which one is the most interesting depending on the use or the application.



**Figure 5.2.** SBSs used in study #2.

In the SBS graph (figure 5.2) it can be appreciated that the viscosity behavior with shear rate is slightly different. However, the four raw materials are in the same range, around 10000Pa·s at zero shear rate.

## 5.2. Initial assessment of LLDPE + SBS blends

### 5.2.1. Description

The second study, which is the most extensive, is presented more deeply.

#### **Study #2**

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**Title:** Initial assessment of LLDPE + SBS blends.

**Description:** Blown monolayer blends of Styrene-Butadiene-Styrene (SBS) copolymer with LLDPE.

**Purpose:** Analyze the behaviour of SBS blended with LLDPE.

---

This study is a preliminary study. Its value is not the samples as such, but their properties. Before manufacturing many different samples, it is better to spend some time studying potential mixtures using different extrusion conditions such as orientation, temperature profile, output, etc. Here the reliability is very important because this step is the base above which the main study is developed.

The raw materials used in this study are the analyzed materials in the first study (section 5.1.1) plus one compatibilizer and one LDPE. The compatibilizer used is maleic anhydride grafted polymer using a base of a high density polyethylene (PE-g-MAH). It is a functional polymer with a density of 0.96 g/cm<sup>3</sup> with a melt index (MI) of 2.0 g/10 min.

In contrast, the LDPE used is fractional melt index Low Density Polyethylene resin with a density of 0.926 g/cm<sup>3</sup> and a MI of 1.0 g/10 min. It presents excellent optical properties which can be observed in LLDPE blends. It improves the optics in LLDPE blends, though it worsens the mechanicals.

Taking as independent variables, we are going to study: the effect of LLDPE or SBS type, of premix, the addition of LDPE, the SBS concentration, the output and the temperature profile. The idea is analyze which variables have more influence in the extrusion process and extract some conclusions from the results so as to know which of them must be taken into account. The study is composed by 16 samples which are shown in the following table with their extrusion conditions.

**Table 5.1.** Samples with extrusion conditions of study #2.

Sample ID	base resin	SBS	Output	[SBS]	Premix Buss	Compatibilizer	20% LDPE	Extrusion Temperature
1	LLDPE A	SBS 1	Low	15%	yes	no	no	Low
2	LLDPE B	SBS 1	Low	15%	yes	no	no	Low
3	LLDPE A	SBS 1	Low	5%	yes	no	no	Low
4	LLDPE B	SBS 1	Low	5%	yes	no	no	Low
5	LLDPE A	SBS 1	Low	15%	yes	no	no	Low
6	LLDPE A	SBS 1	High	15%	yes	no	no	Low
7	LLDPE A	SBS 1	Low	15%	no	no	no	Low
8	LLDPE A	SBS 1	Low	15%	yes	yes	no	Low
9	LLDPE A	SBS 1	Low	15%	yes	no	yes	Low
10	LLDPE A	SBS 1	Low	15%	yes	no	no	High
11	LLDPE A	SBS 2	Low	15%	yes	no	no	Low
12	LLDPE B	SBS 2	Low	15%	yes	no	no	Low
13	LLDPE A	SBS 2	Low	5%	yes	no	no	Low
14	LLDPE B	SBS 2	Low	5%	yes	no	no	Low
15	LLDPE A	-	Low	-	-	no	no	Low
16	LLDPE B	-	Low	-	-	no	no	Low

When paying attention to the samples' conditions, it can be observed that the 1<sup>st</sup> and 5<sup>th</sup> samples have the same composition in the same conditions. These two samples were duplicated in order to compare them to one another as a way to estimate the reproducibility of the results. And there are two "zeros" so as to compare all the samples; the zeros being the reference pure LLDPEs that is to say samples 15 and 16.

As it is explained in section 3.1.1, working with 16 samples is complex and it is carried out as a measure to simplify the manufacture and be more time-effective. The samples which required a premix in the Buss Compounder Line were grouped by composition. Afterward, 4

masterbatches were manufactured with a higher concentration, so taking a part of them and adding the corresponding LLDPE, and in some cases LDPE or/and PE-g-MAH, the samples are obtained by diluting up to specification.

Sections 3.1.1 and 3.1.4 include the composition of the MBs and all the parameters related to the extrusion process. Once the mixtures are done, they are introduced into the extruder through the hopper, where the mixture is melted.

Before extruding them it is very important to know the properties and the rheology of the raw materials. Therefore, in the following section a rheological study is presented.

In this study the polymers used have a different density and a very different melt index. The idea is to observe their behaviour and decide which ones are the best materials to combine.

### 5.2.2. Rheological study

The pure components have one viscosity in function of the shear rate as it is shown in figures 5.1 and 5.2. However, the resulting blend does not follow a lineal correlation. The blends follow a logarithmic tendency and one correlation which can describe well the tendency is the next:

$$\log \eta = v_1^2 \cdot \log \eta_1 + 2 \cdot \tau \cdot v_1 \cdot v_2 \cdot (\log \eta_1 \cdot \log \eta_2)^{1/2} + v_2^2 \cdot \log \eta_2 \quad (5.1)$$

Where  $\tau$  is a parameter which can be calculated experimentally or with the next expression in some blends.

$$J = 0.53613 + 0.72537 \cdot R - 0.21682 \cdot R^2 \quad (5.2)$$

Understanding  $R$  as the logarithmic quotient between the two components.

$$R = \frac{\log \eta_1}{\log \eta_2} \quad (5.3)$$

Defying  $v_i$  as volume fraction.

$$v_i = \frac{x_i \cdot \rho_i + x_{i+1} \cdot \rho_{i+1}}{x_i \cdot \rho_i} \quad (5.4)$$

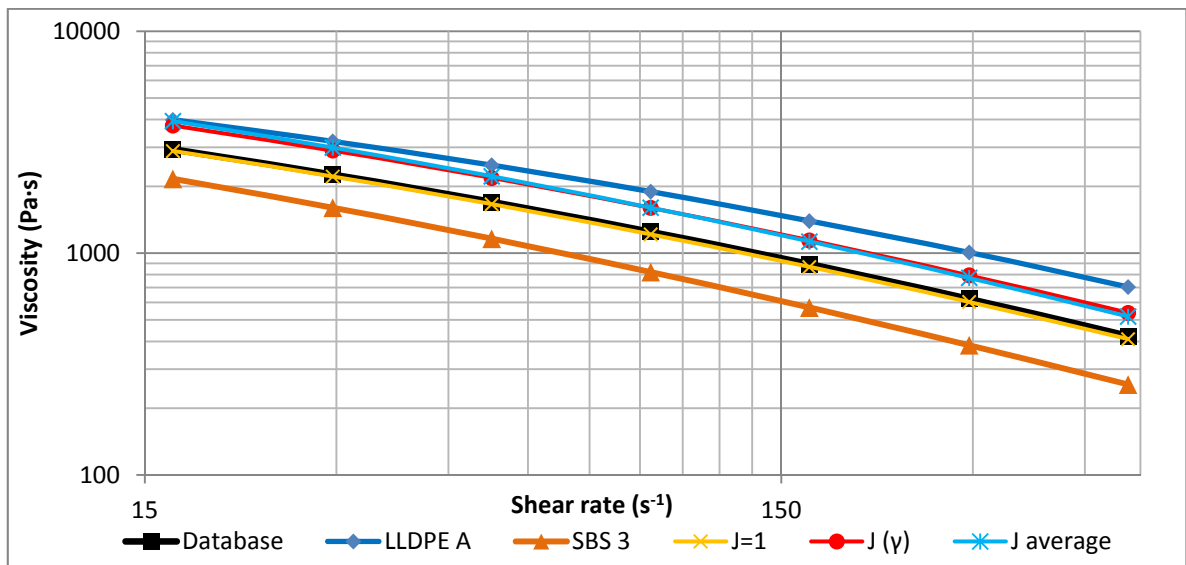
In the consulted report<sup>12</sup> appears that the correlation obtained good results predicting the blends which appear below.

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<sup>12</sup> Victoria Dobrescu, "An Empirical Model for Melt Viscosity of Polymer Blends". Institutul de Cercetări Chimice-ICECHIM, 202, Splaiul Independentei, Bucharest, Romania. Editorial Polymer Bulletin, Springer-Verlag 1981.

- HDPE/HDPE
- HDPE/LDPE
- LDPE/LDPE
- HDPE/PP
- HDPE/SBS
- PS/SBS

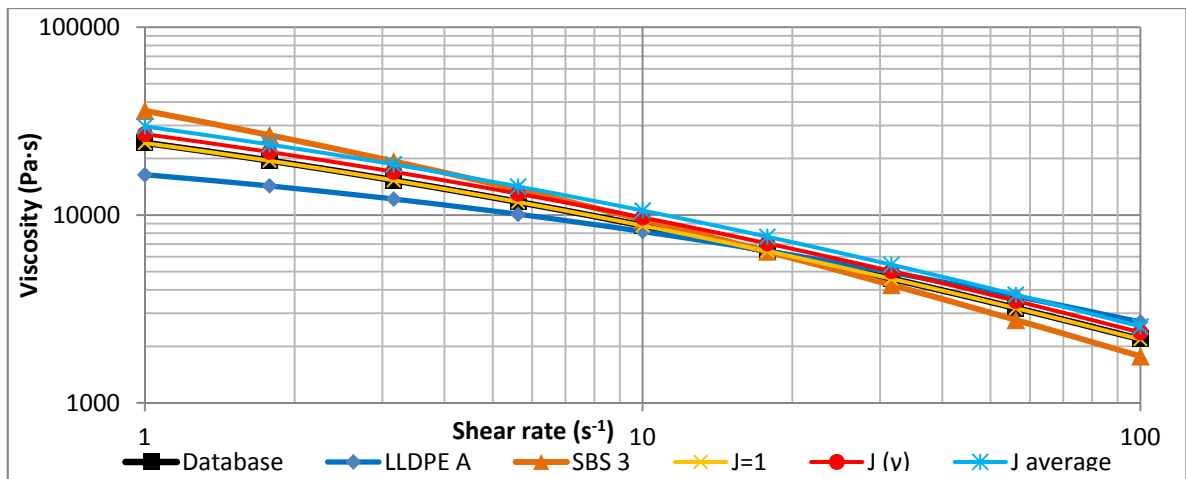
Another option is takes  $J = 1$  or determines the average value. In the next graph is shown the three hypotheses. In the following graph it is shown a comparison of correlations in a mixture of LLDPE + SBS, where the LLDPE is the same as LLDPE A and SBS is a very similar SBS which is included in the database.



**Figure 5.3.** Correlation's hypothesis for a blend with 50% of LLDPE A and 50% of SBS 3 at 190°C.

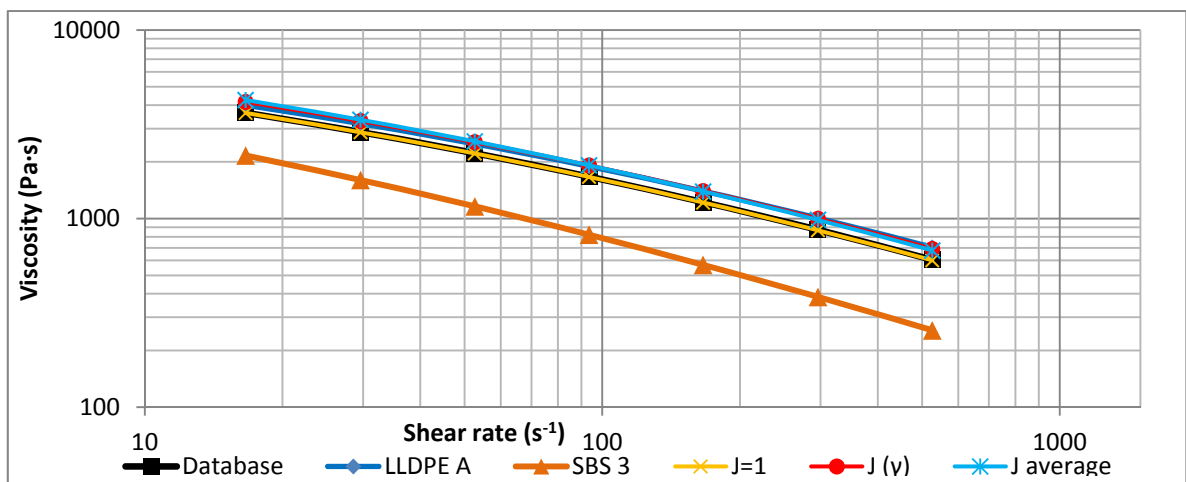
Comparing the three trends with the database curve, the  $J(\gamma)$ , where  $J$  is with regards to shear rate ( $\gamma$ ), is higher, after goes the correlation using the average of  $J$  values which is a bit higher than  $J(\gamma)$  at the beginning and a slightly lower at the end. Finally, the hypothesis which predicts better the behaviour is considering  $J = 1$ . This is the best prediction, therefore, this hypothesis is accepted and the following graphs are doing taking this hypothesis as a true.

The next step is study the effect of temperature and fraction in the estimated values. In the next graphs it is represented the same blend with the same fractions, but at 150°C. Maybe the differences between values are slightly lower, but the better hypothesis is the same. However, the temperature affects more than shear rate, the curves presents higher different when the test temperature changes than when the shear rate varies.



**Figure 5.4.** Correlation's hypothesis for a blend with 50% of LLDPE A and 50% of SBS 3 at 150°C.

Varying the fraction the result is the following:



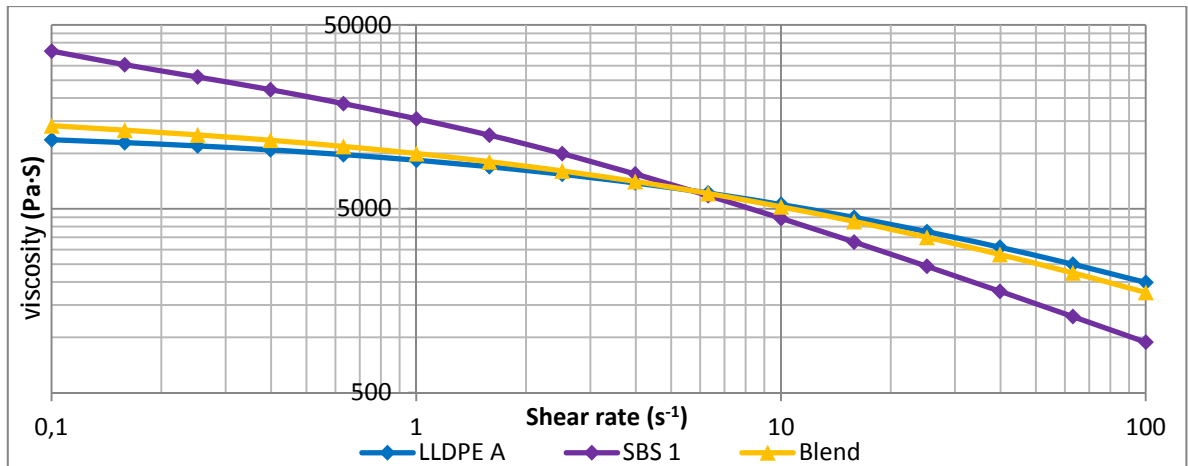
**Figure 5.5.** Correlation's hypothesis for a blend with 85% of LLDPE A and 15% of SBS 3 at 190°C.

In the figure 5.5 can be observed that the effect of increase one fraction implies a slightly tendency to decrease the differences. But the error continues to be enough high to take into account and it cannot be neglect. In conclusion, at high temperatures tends to predict worst using the expression 5.2 and 5.3 or doing the average of the values. At the same time, when the more difference in component fractions the worst predict. Therefore, at lower temperatures and equal fractions predicts better, but the well prediction and the simplicity of take  $J$  as 1 lead to accept that hypothesis.

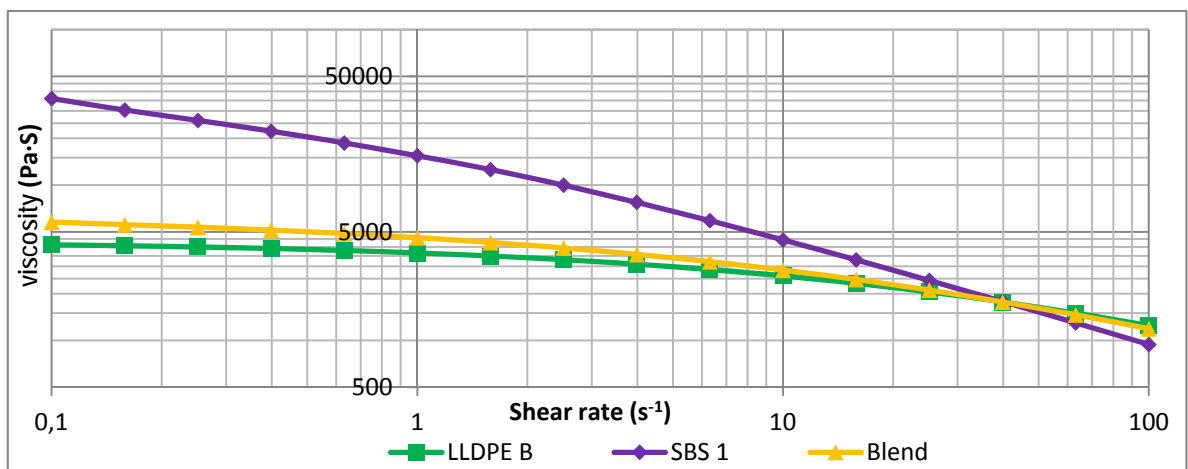
Taking this consideration, the values offer by the correlation, taking  $\tau$  as 1, define better the shear rate comparing with the values of database, therefore this correlation offers a good results to the next blends:

- LDPE/LLDPE
- LLDPE/SBS

In the following figures, it can be appreciated the mentioned tendencies at 190°C.



**Figure 5.6.** Viscosity curve versus shear rate in a mixture of 85% of LLDPE A and 15% of SBS 1.

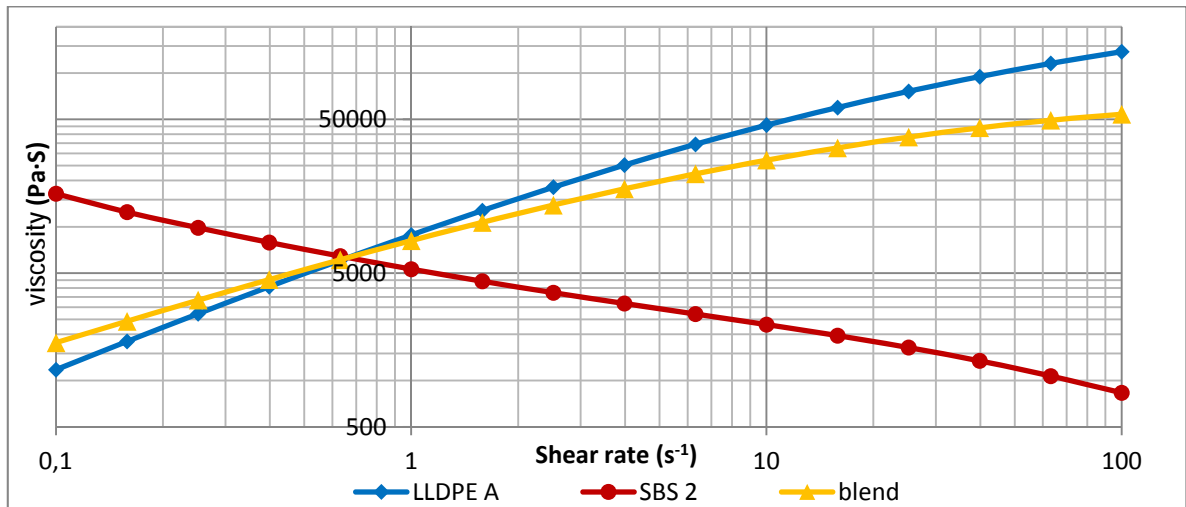


**Figure 5.7.** Viscosity curve versus shear rate in a mixture of 85% of LLDPE B and 15% of SBS 1.

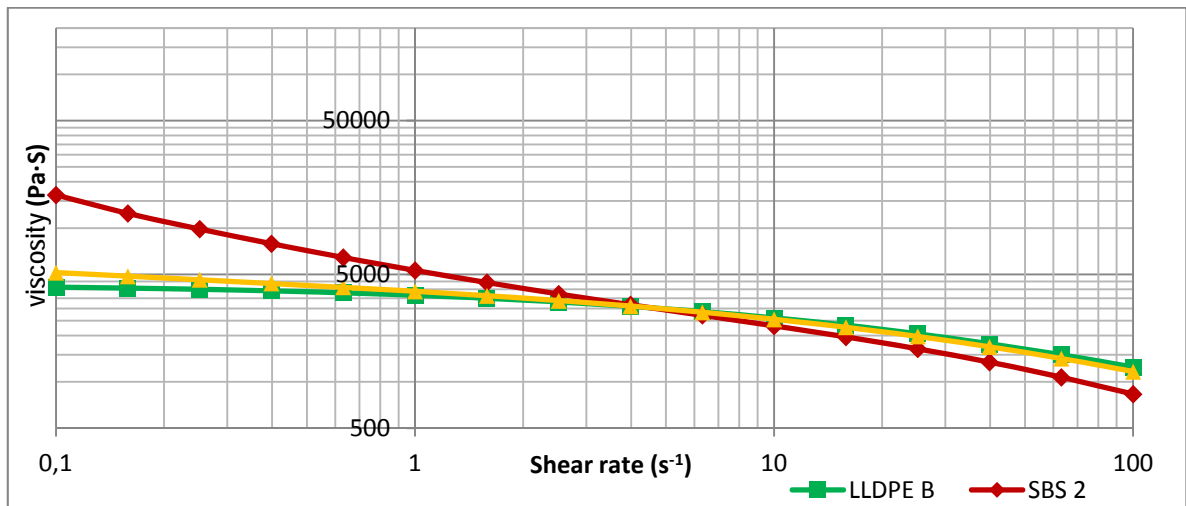
The two LLDPEs present a different viscosity at shear rate zero. They were selected because they have different properties and the idea was identify which one presents better properties when mixed with SBS. In these two graphs the SBS is the same, and the variation in viscosity values exists only in case of LLDPE. It can be observed that LLDPE A offers the highest viscosity which is the same as SBS 1 at a lower shear rate, about  $6,5 \text{ s}^{-1}$ . In contrast, in figure 5.7 it is shown at  $13 \text{ s}^{-1}$ . The difference is nearly twice as it was identified in figure 5.1 comparing the two LLDPEs.

This behavior implies that the mixture with LLDPE A will present a higher viscosity if both blends are extruded in the same conditions. Viscosity is the most important flow property which is basic in extrusion to understand the processing window. It is indispensable to know the viscosity curve of the used resin, versus shear rate, because if not the processor has no idea about the relation between viscosity and shear rate and the temperatures used in the processing. In all the figures, there is a point where the curves cross. For example, in figure 5.8, SBS 2 presents a high viscosity and LLDPE a lower one, and they cross at one point. From

that point onwards, the viscosity effect changes. The tendency varies depending on the processing procedure and the location of the viscosity process curve.



**Figure 5.8.** Viscosity curve versus shear rate in a mixture of 85% of LLDPE A and 15% of SBS 2.



**Figure 5.9.** Viscosity curve versus shear rate in a mixture of 85% of LLDPE B and 15% of SBS 2.

In reference to study 212749, viscosity with regards to shear rate is also critical in coextrusion process. The presence of interfacial instabilities is very common and to prevent them among adjacent polymer layers, it is essential that the resins have the same viscosity in the die and/or feed block because here is where they are melted.

Therefore, that is the specific point around which the resins present similar viscosity values. Selecting the appropriate extrusion conditions is very important and in this case the reason is to avoid interfacial instabilities. Very different viscosities could create instabilities during the extrusion process. But, preferably the shear rate should be estimated previously and then determine the temperature at which the resins have the same viscosity.

### 5.2.3. Results & discussion

In section 5.2.1 are shown the extrusion conditions. The samples are produced and conditioned during 48 hours in the laboratory, and then they are tested. The properties which are tested are the following:

- Impact, Puncture and Tear Resistance.
- Secant Modulus and Tensile Stress.
- Clarity, Gloss and Haze.
- Seal Strength.
- Permeability.

In the sections below are presented the obtained results after test the mentioned properties with the corresponding test equipment.

#### 5.2.3.1. Mechanical properties

The tested mechanical properties are:

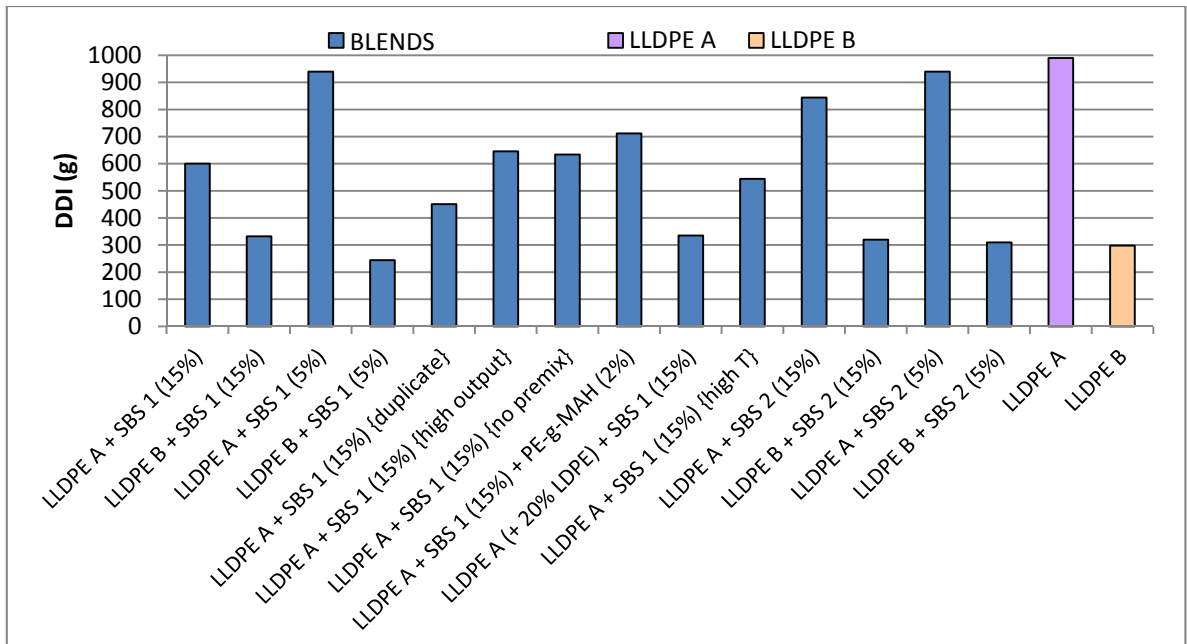
**Table 5.6.** Tests associated with the properties.

<b>Properties</b>	<b>Test</b>	<b>Film direction</b>
Impact resistance	Dart Drop Impact	MD
Puncture resistance	Puncture	MD
Tear resistance	Elmendorf	MD <sup>13</sup> and TD <sup>14</sup>

One important parameter which must be taken into account before starting the tests is the film direction. In some tests it is not important, in others it is vital. When a blown film is extruded, the nip rolls pull at the film in MD. This fact increases the orientation grade in MD because the molecules are more orientated. In contrast, in cast films, there are no rolls pulling at the film which falls and is collected in the rolls, in such case the orientation is more balanced. In this case, neither impact nor puncture resistance require to be specified the direction because the film receives the same force in both directions. However, tear resistance is influenced by molecular orientation. In Elmendorf test the intermolecular bonds are broken with higher or lower tear strength depending on the tear direction. In the following figures the results of the tests, mentioned in table 5.6 are shown.

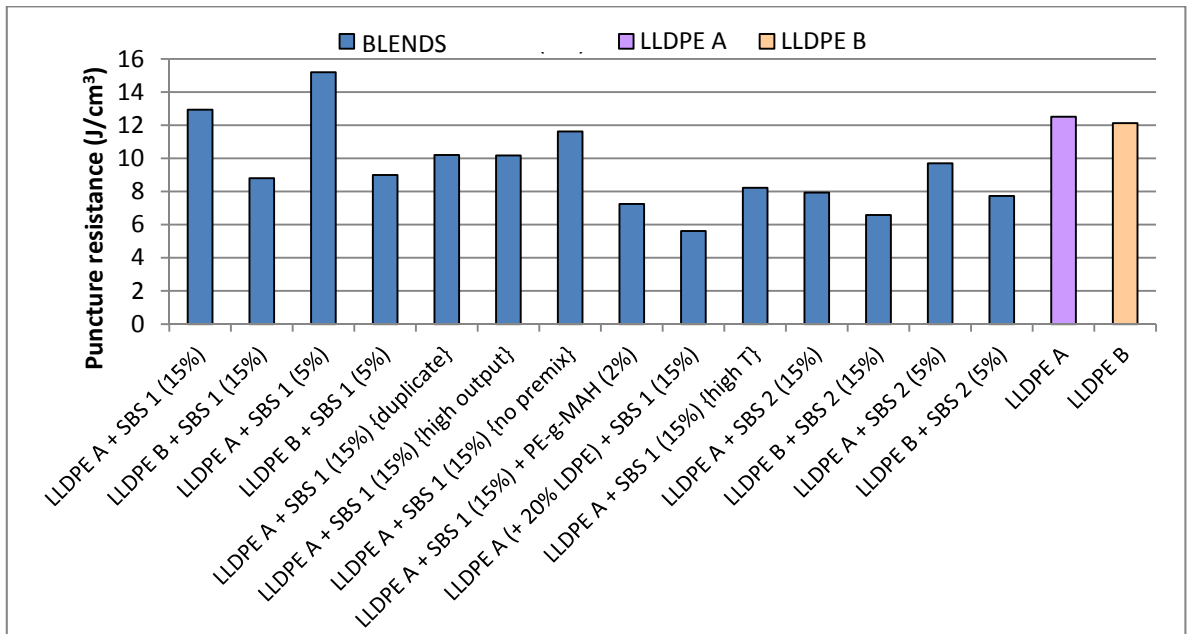
<sup>13</sup> **Machine Direction (MD).** The direction of the film when is pulled to the rollers.

<sup>14</sup> **Transverse Direction (TD).** It is the perpendicular direction to the bubble direction.



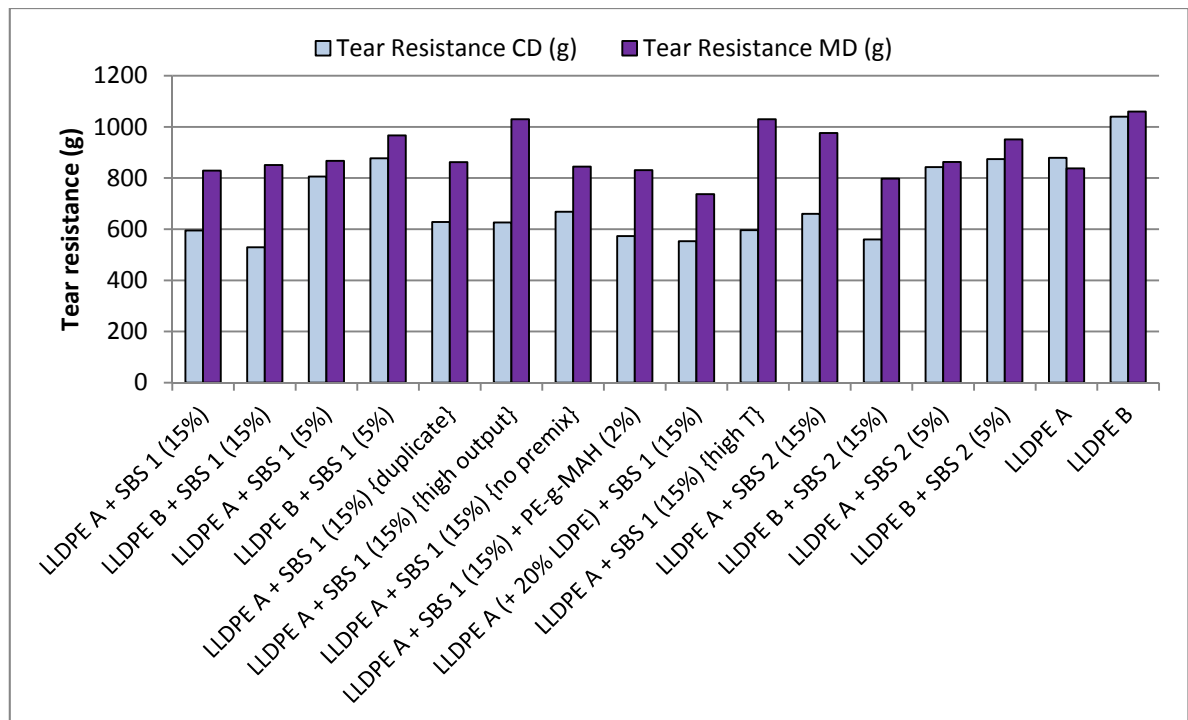
**Figure 5.10.** Dart Drop Impact results.

Figure 5.10 shows how adding 5% of SBSs, LLDPE A blends stay the same dart drop impact; otherwise, adding more decreases a lot. And in case of LLDPE B, it stays the same in all cases.



**Figure 5.11.** Puncture results.

Puncture results graphs show how LLDPE B goes down with any blend, adding SBS 1 or SBS 2. Comparing to the pure LLDPE B, the blends always have a lower puncture resistance. In contrast, amongst LLDPE A blends, some samples with SBS 1 present improvements to the pure LLDPE A. Adding 5% or 15% of SBS, the grade of improvement change. However, the blend with 5% improves significantly and has to be checked in the following study.



**Figure 5.12.** Tear resistance results.

In Elmendorf results can be appreciated that the values suggest very little film orientation for pure LLDPEs and, with blends, there are two tendencies. In MD tear goes down and in TD tear holds or, in some cases, increases. Pure samples present a well-balanced tear; it is nearly the same in both directions. In all the situations, adding 15% of SBS imply important decreases and the blends with a 5% of SBS tend to maintain the best balance.

It is well-known that the presence of larger tear strength in TD than in MD one. This is caused because in these films have higher orientation in MD than in TD. In MD, the crack can initiate and propagate between lamellar stacks, resulting in lower energy required for tear propagation. In TD tear the orientation is not favourable and for this reason is dissipated more energy during the crack propagation.

In case of LDPE, it is more sensible to processing condition. However, they have very small effect. Higher die gaps leads to higher MD and TD tear strength, but higher output has not effect in MD and TD is reduced. LDPE films present a row nucleated structure with twisted lamellae along TD resulting a strong plane. This is responsible that the tear strength in MD is higher than in TD.

However, the logical tendency should be that MD stabilizes and TD tends to increase, but the extrusion line used does not orientate as it should do; it is too soft.

### 5.2.3.2. Elastic and plastic deformations

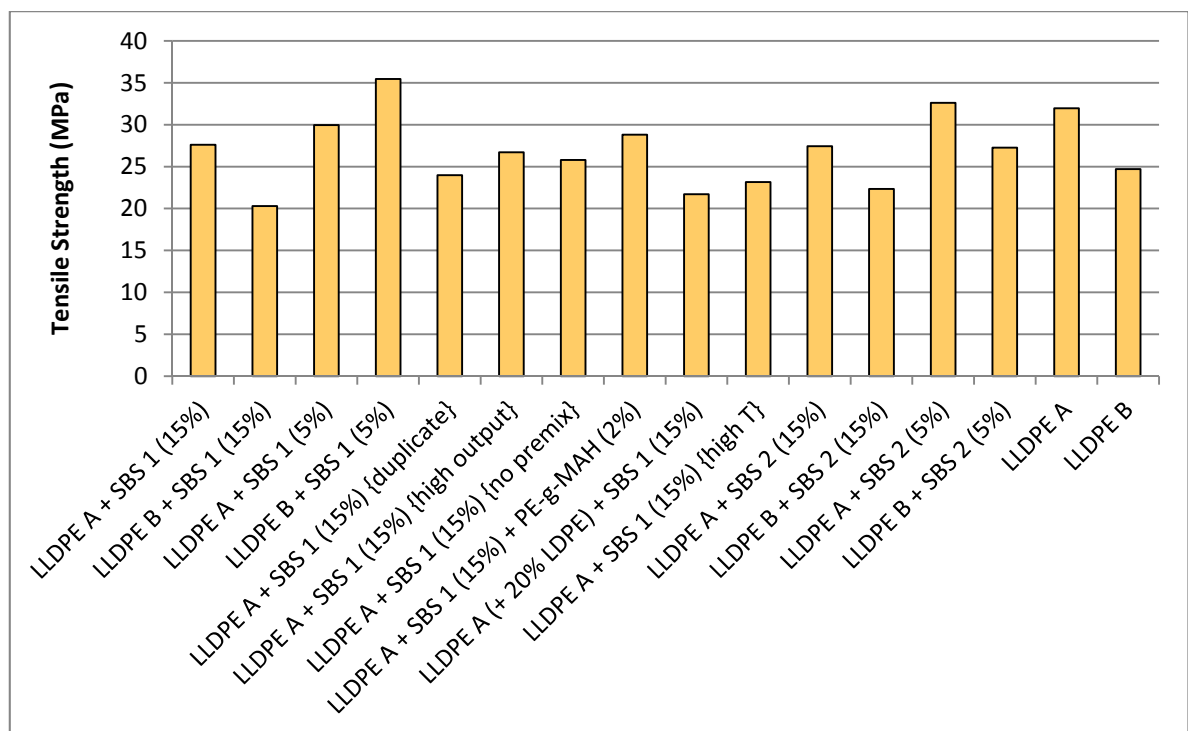
Elastic and plastic deformations are two important parameters when it is necessary to characterize a film. In the following table can be appreciated the elaborated tests and the properties which offer, obviously, elastic and plastic deformations are included.

**Table 5.7.** Plastic properties associate with deformation.

Test	Properties	Film direction
Tensile	Tensile strength	TD
	Toughness	TD
Secant	Secant Modulus	TD
	Young Modulus	TD

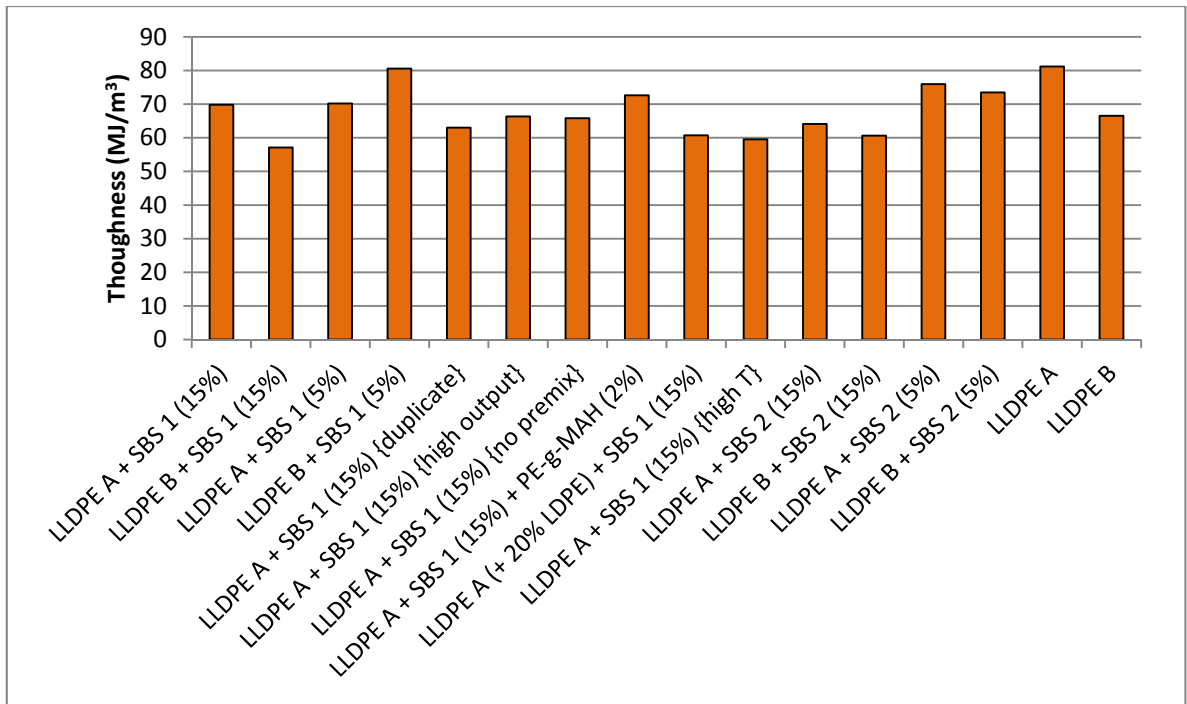
In this section are presented the results obtained of the tests mentioned in the table. These tests are done by a robot which is the responsible of provide the results. Once the robot finish, the results are check and if they do not satisfy the specification are repeated.

In the following two graphs are shown the results of Tensile Test.



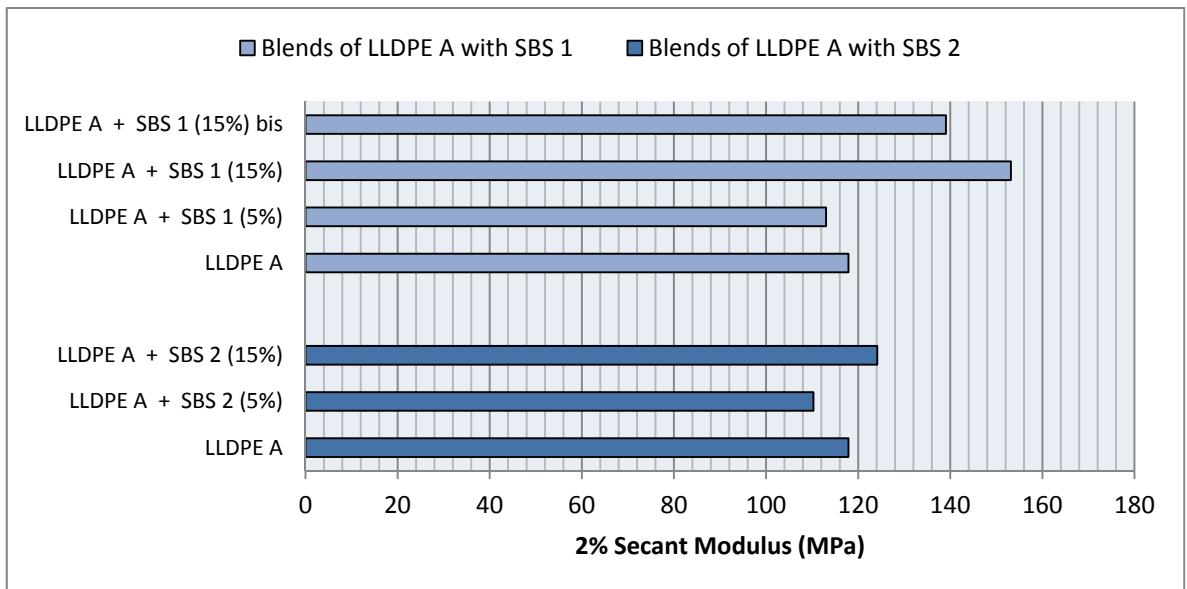
**Figure 5.13.** Tensile strength results in TD.

In general, observing figure 5.13 there no evident improvement. Adding different % of SBS there no tendency, all the results are between 20 and 35 MPa. Indeed, most of them between 22 and 30 MPa. In this case, a difference of 5 or 8 MPa, when the acceptable deviation corresponds to the 10%, there are no important improvements.



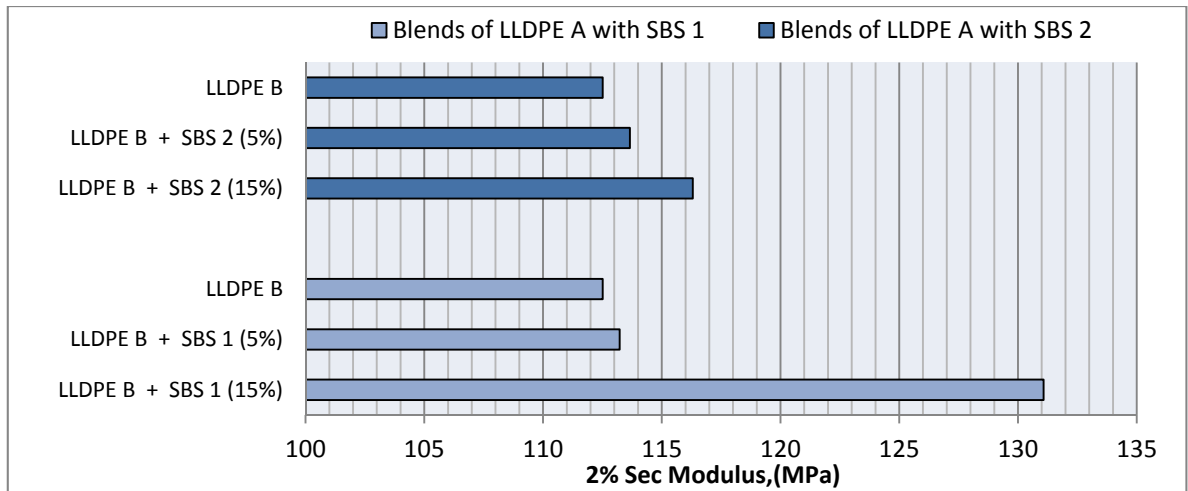
**Figure 5.14.** Toughness results in TD.

In case of toughness, the observations are the same. Most of the values are between 60 and 75. The differences are not significant enough to be taken into account. There is no tendency to analyse. The results of the secant test are presented in the following figures.



**Figure 5.15.** Secant Modulus (2%) in TD for blends with LLDPE A.

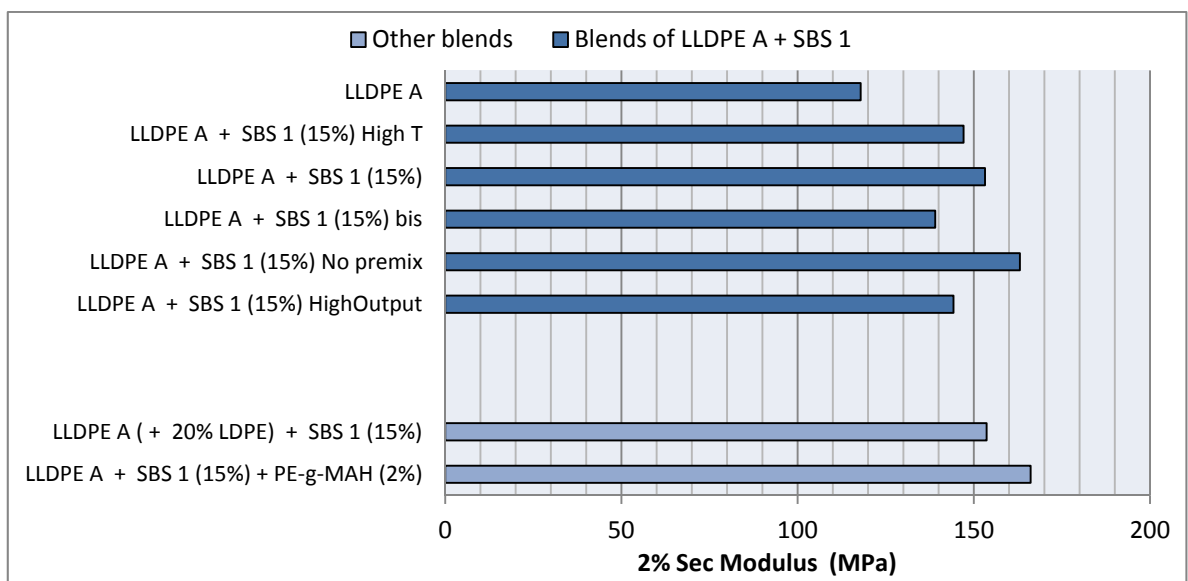
The different blends of LLDPE A are compared in the figure above. For blends with SBS 1, the 15% improve the pure sample, but the 5% does not. There is a duplicate of blend with 15% and, although the results are not the same, they are similar and in both cases an improvement can be observed compared to the pure LLDPE A. In contrast, in blends with SBS 2, there is nearly no improvement or worsening. Neither adding 5% nor 15% of SBS 2.



**Figure 5.16.** Secant Modulus (2%) in TD for blends with LLDPE B.

Blends with LLDPE B are shown in figure 5.16. The result is the same as in figure 5.15. The effect of SBS 2 is not significant and the SBS 1 affects more widely if the used percentage corresponds to the 15%, lower than this the improvement is negligible. One important difference is that the experimented improvement of pure LLDPE B is much higher than that experimented by LLDPE A.

And finally, a graph, where the fix reference is LLDPE A + SBS 1, is presented. When the study was defined it was supposed that this blend could be an interesting sample to study. It is known that SBS produce important improvement in the mechanical properties of some materials; for these reason, one of the LLDPEs and one SBC were selected in order to extrude the same sample changing the conditions. The results of the samples extruded and the two samples with LDPE and PE-g-MAH are shown in the following graph.



**Figure 5.17.** Secant Modulus (2%) in TD for blends with LLDPE A.

It can be appreciated that in all the cases there are significant improvements regarding the pure LLDPE. The combinations which present better improvements are the blend with PE-g-MAH and the blend which was not premixed in the Buss compounder. In contrast, the addition of LPDE does not have any effect and the increase of output and T do not show significant changes, this is because the extrusion line used did not allow to change processing conditions in a wide range and, for this reason, the blends with higher or lower output or T do not present a significant different in properties, like secant modulus.

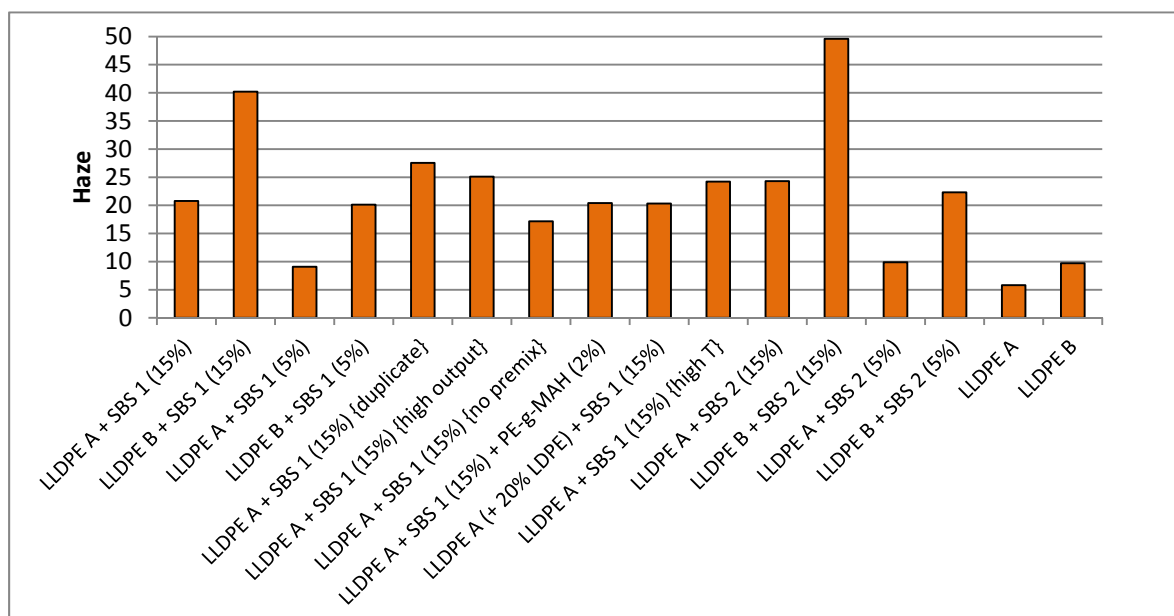
### 5.2.3.3. Optical properties

The optical properties are basic in this study. The idea is to find a well-balanced blend which combines both good optics and good mechanics. The measured optical properties are summarized in table 5.8.

**Table 5.8.** Optical properties measured in order to characterize the blends.

Test	Properties
Haze	Haze
Gloss 45°	Gloss
Clarity	Clarity

In the following graphs the effect of adding SBS can be appreciated. It is known that SBS have an excellent optics, however sometimes it can presents incompatibility effects and the optics worsen <sup>(ref. 11)</sup>. Firstly, the results of the haze test are represented in the next graph.



**Figure 5.18.** Results of Haze.

In this case, one point of haze makes a significant difference; therefore, bigger differences must be taken into account. A good Haze is the lowest possible value, such as 0.9 or 1. LLDPE A presents a better haze than LLDPE B. When compared, the differences are considerable. In

both cases, only the 5% SBS blends are comparable to pure LLDPEs, otherwise the values worsen too much. Focusing on the blends with PE-g-MAH or LDPE, the conclusion is that they do not present improvements significant enough to be considered (ref. 12).

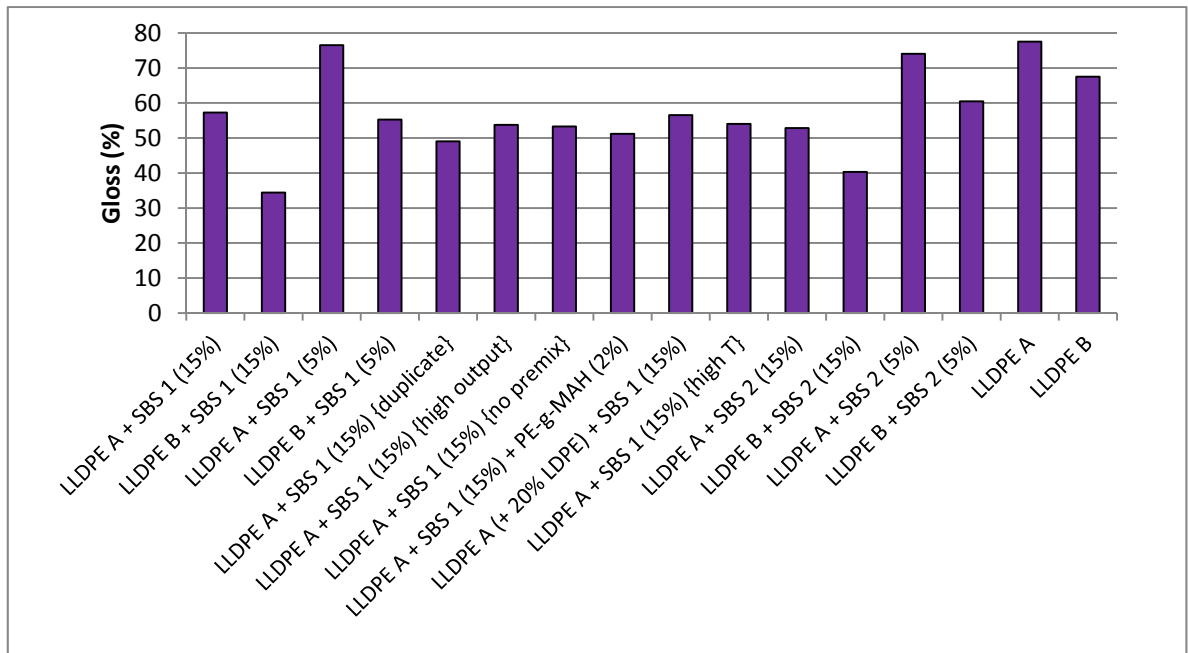


Figure 5.19. Results of Gloss 45°.

Here, the conclusion is the same. There is no blend with a percentage of SBS higher than 5% which can be compared with the pure LLDPEs. Only the blends with 5% of SBS are a realistic option. However, they can be interesting in specific application where the optical properties are not important. And, on that occasion, the effect of PE-g-MAH or LDPE is not significant.

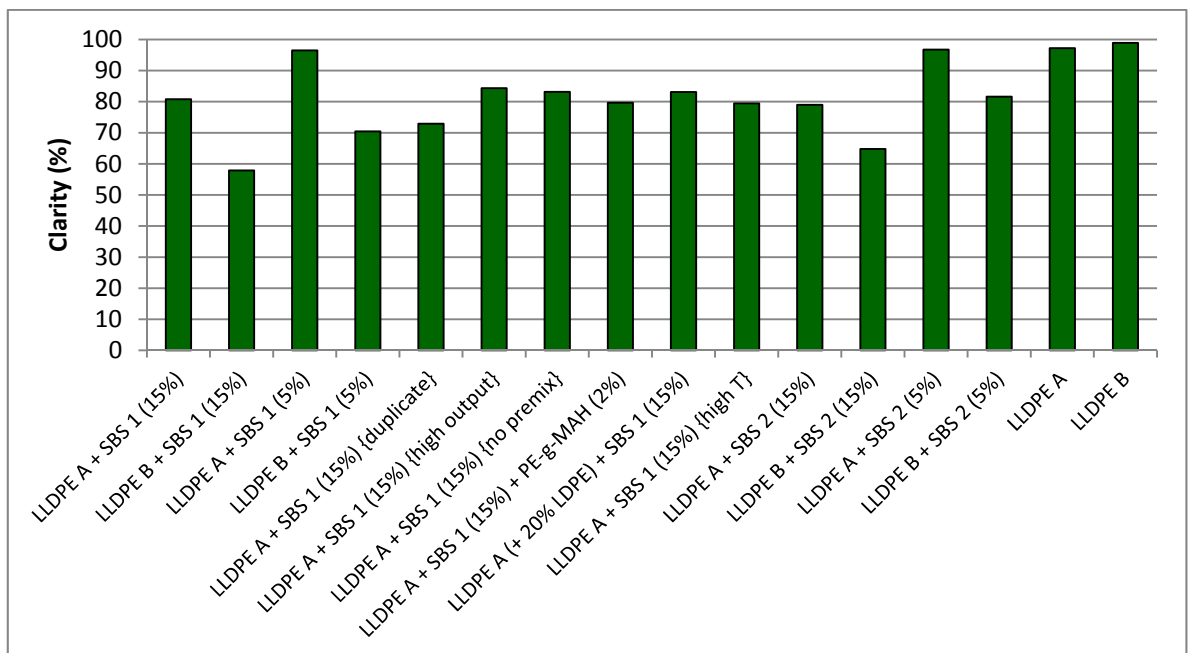


Figure 5.20. Results of Clarity.

Finally, the clarity tendency is the same. In all the measured optical properties the tendency does not change. The only blends which can be accepted are those with 5% of SBS 1 or SBS 2. In general, there are no significant differences in the behavior of the two SBCs used. However, the type of LLDPE is not negligible. In general, LLDPE A presents a better Haze, Gloss and Clarity combined with SBS 1 or SBS 2. Therefore, in terms of optical properties, the LLDPE A blends are the most interesting to study, specifically with 5% of SBS 1.

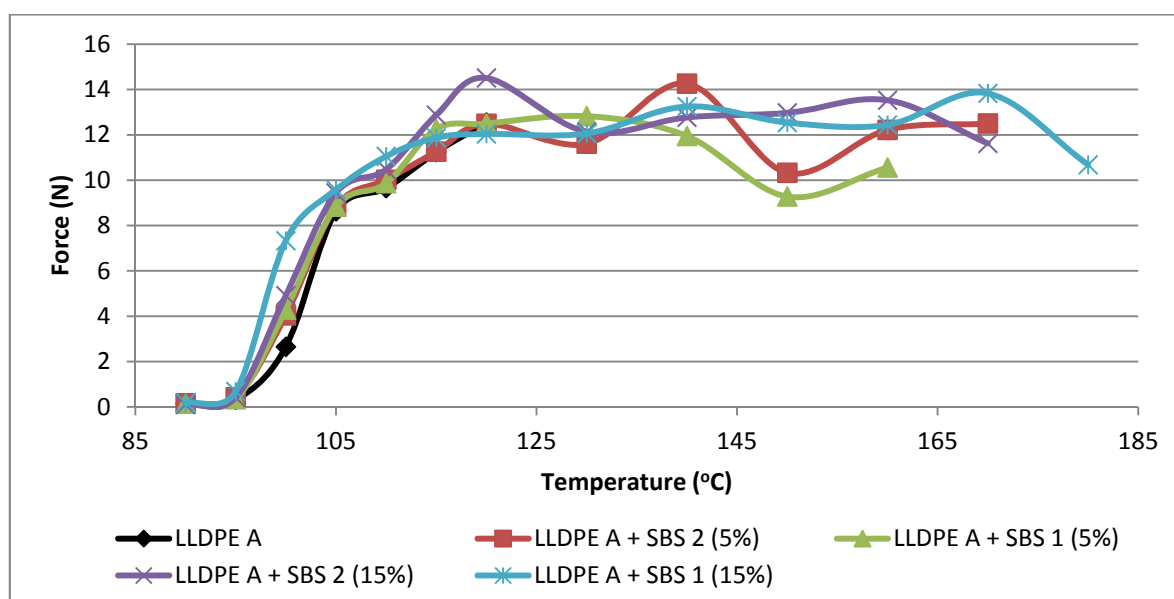
#### 5.2.3.4. Sealability properties

Apart from the properties listed before, another interesting property is sealability. In this study two different tests are used to determine the sealing capacity of the blends. The tests, which appear in the table 5.9, differ both in the preparation of the samples and their conditions during the test.

**Table 5.9.** Seal Strength tests.

Test	Properties	Film direction
Seal Strength Test	Seal Strength	TD
Hot Tack Test		TD

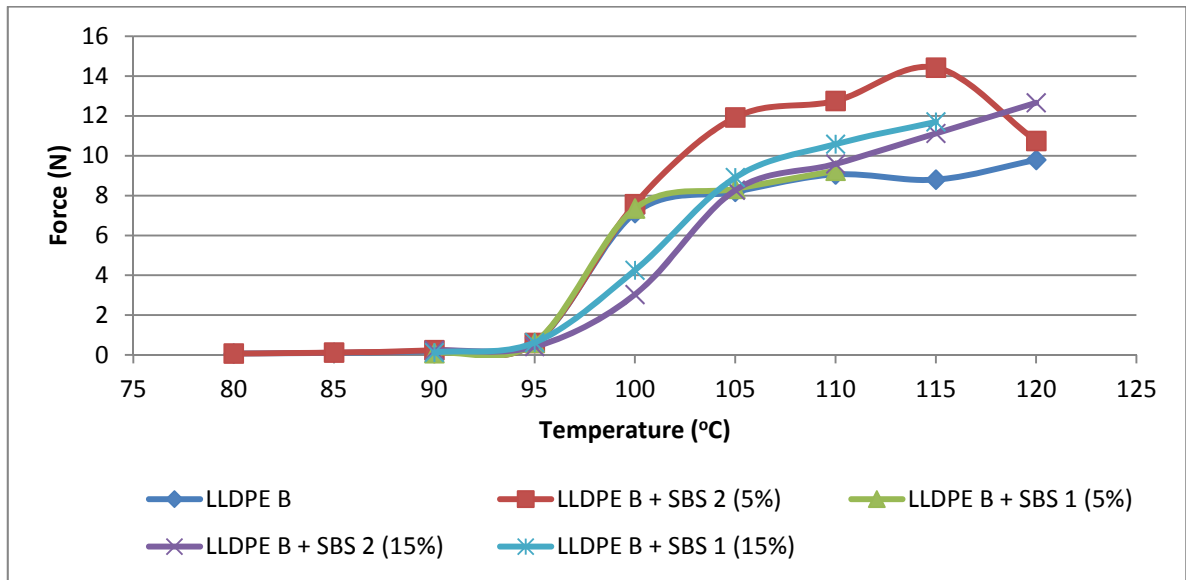
Seal Strength<sup>15</sup> is interesting to evaluate in order to determine the opening force used to choose the most appropriate plastic to produce consistent seals. In case of Seal Strength, the samples have been prepared 48 hours before and the test is carried out at laboratory environmental conditions. The results of this test are shown in the following 4 graphs.



**Figure 5.21.** Seal Strength results of the blends of LLDPE A.

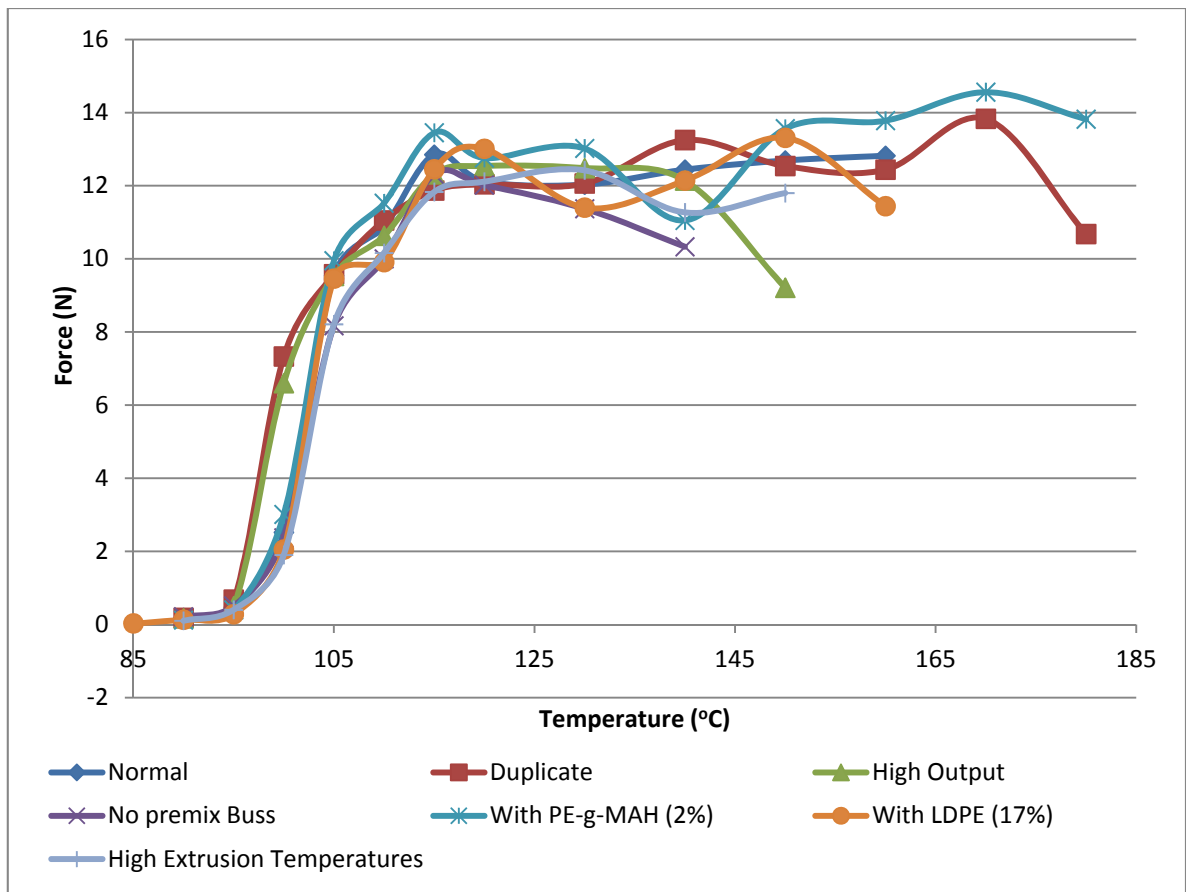
<sup>15</sup> It measures the strength of seals within flexible barrier materials. It is a quantitative measure for use in process validation, process control and capability

It can be observed that all the blends present a broader seal plateau in comparison with the pure LLDPE A which is represented by the black curve in figure 5.21. The blends can be sealed at 40 to 60°C more than the pure LLDPE. In terms of seal strength, there are no significant differences.



**Figure 5.22.** Seal Strength results of the blends of LLDPE B.

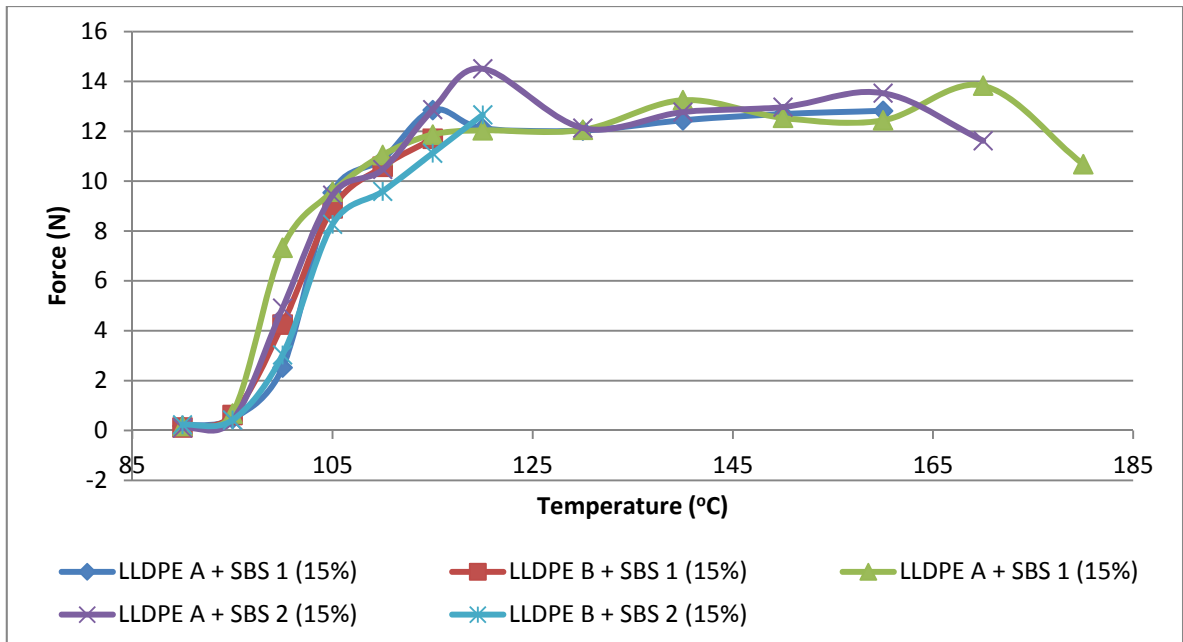
For LLDPE B, the tendency is similar, but there are no evidences of broader seal plateau. Comparing the blends with the pure LLDPE, no significant differences can be appreciated because, in all the cases, the blends can be sealed up to 115 - 120°C like the pure LLDPE and, in terms of seal strength, the force is nearly the same ( $\pm 4$  N).



**Figure 5.23.** Seal Strength results of the blends of LLDPE A + SBS 1 (15%) extruded in different conditions.

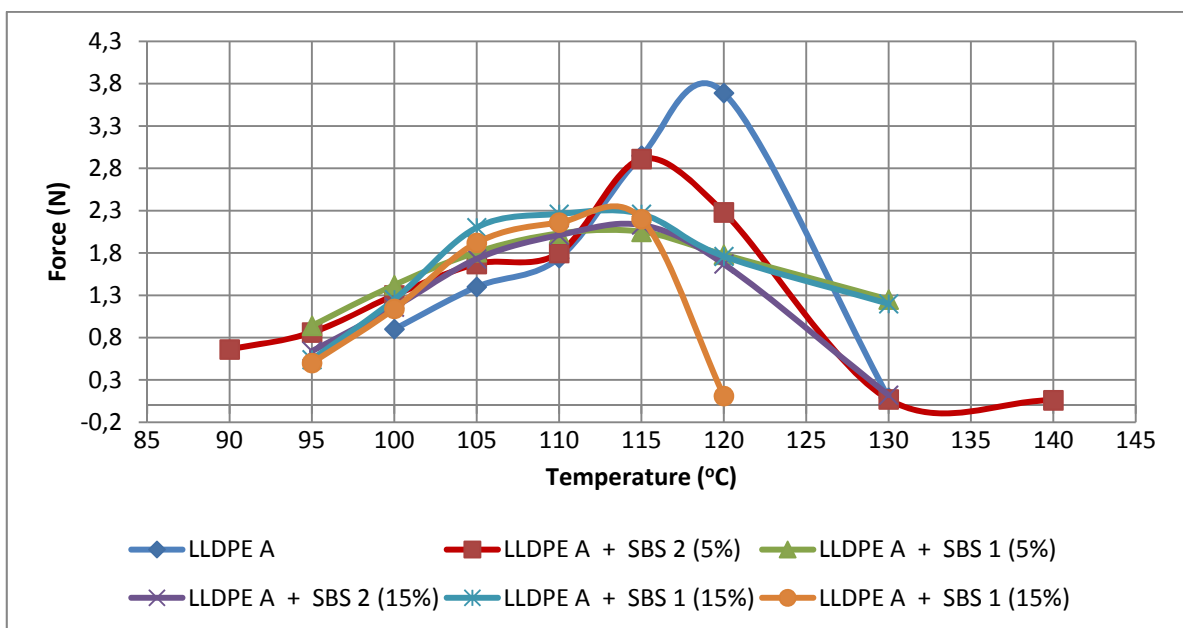
Comparing the tendencies of the same blends, but in different conditions can be observed that the blend which was not premixed has the lowest maximum sealing temperature. At the same time the blends extrude in high output and high extrusion temperatures are not much better. Finally the others present the higher maximum sealing temperature, specially the blends in standard conditions and the blend with PE-g-MAH. However, there are no evidences of clear improvements; it is required to study them more deeply.

Finally, in figure 5.24 are represented the blends with a 15% of SBS independently of the LLDPE or SBS used, all are included.



**Figure 5.24.** Seal Strength results of the blends of SBS (15%).

Comparing these blends, it can be appreciated that LLDPE A blends arrive at much higher sealing temperatures; the difference is about 60°C. Amongst the three LLDPE A blends, although one of them resists more than the others, there are no significant differences between the blends with SBS 1 or SBS 2; as before, it requires to be studied more deeply. In contrast, in case of Hot Tack test, the samples are sealed and tested immediately, to replicate the conditions in industrial form-fill-seal packaging machines. The tester seals the sample and then stretches it measuring the force-elongation curve. The results are shown in the next four graphs.



**Figure 5.25.** Hot Tack results of the LLDPE A blends.

The first graph shows all the combinations with LLDPE A. The pure LLDPE seals resist a higher force than blends with SBS, but the blends seem to be that may decrease initiation temperature; they can be sealed at lower temperature.

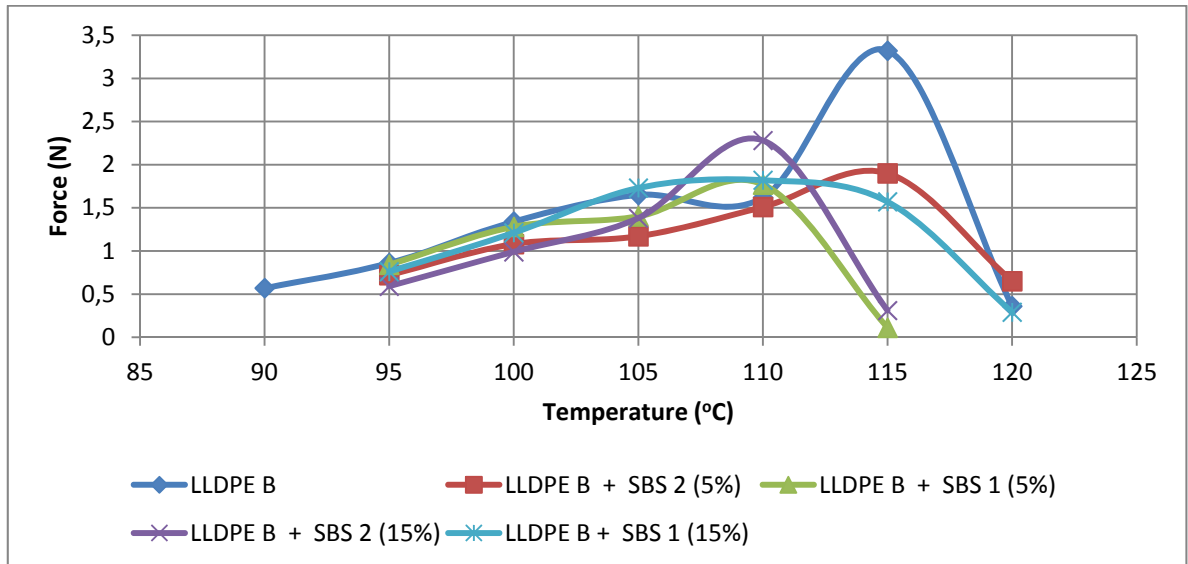


Figure 5.26. Hot Tack results of the LLDPE B blends.

The behaviour observed in LLDPE A blends is not reproduced in LLDPE B blends. In this case, the pure LLDPE presents a lower initiation temperature. However, one common point is the fact that both LLDPEs present the higher force resistance. It seems to be that the addition of SBS may decrease the sealing force, but it permits sealing at lower temperature.

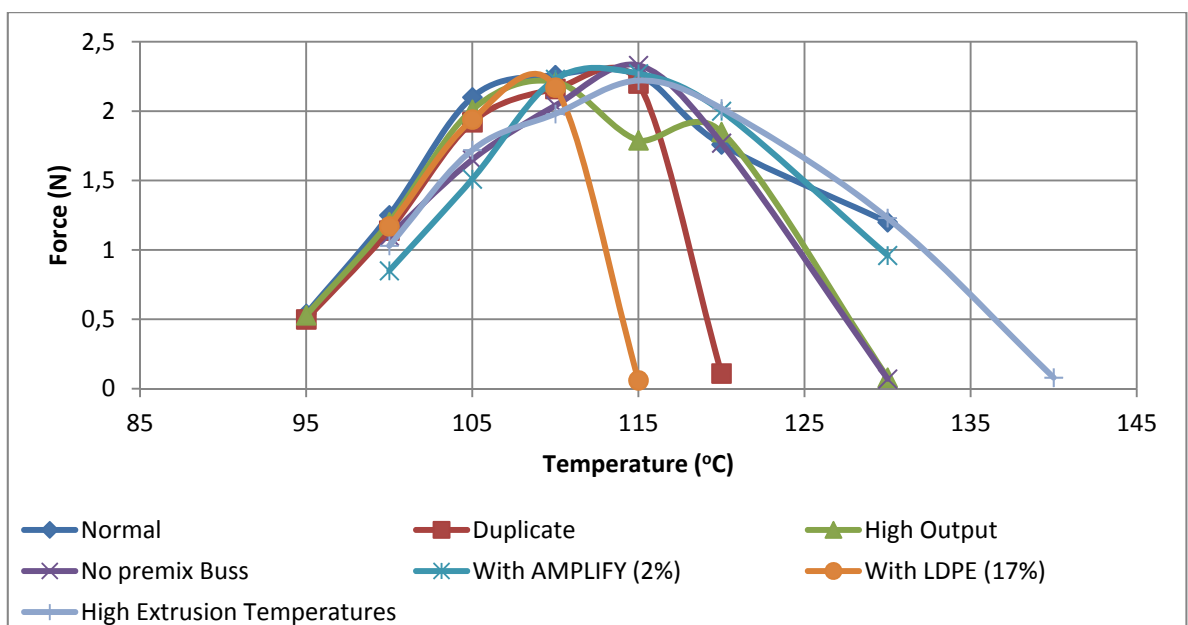
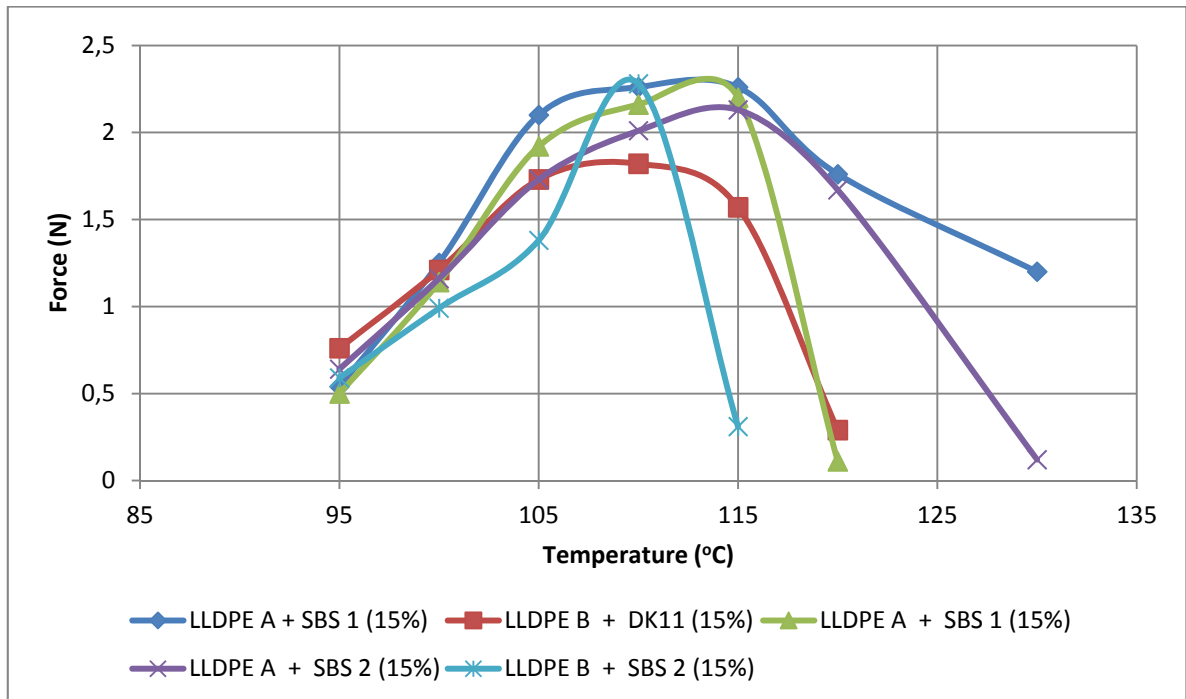


Figure 5.27. Hot Tack results of the blends composed by LLDPE A +SBS 1 (15%).

Analyzing the extrusion conditions, it can be observed that, apart from the high output conditions case, the seal initiation temperature increases in all the cases. However, the maximum force resistance is always the same.

There is not a clear tendency; the graph presents a big variability.



**Figure 5.28.** Hot Tack results of the blends of SBS (15%).

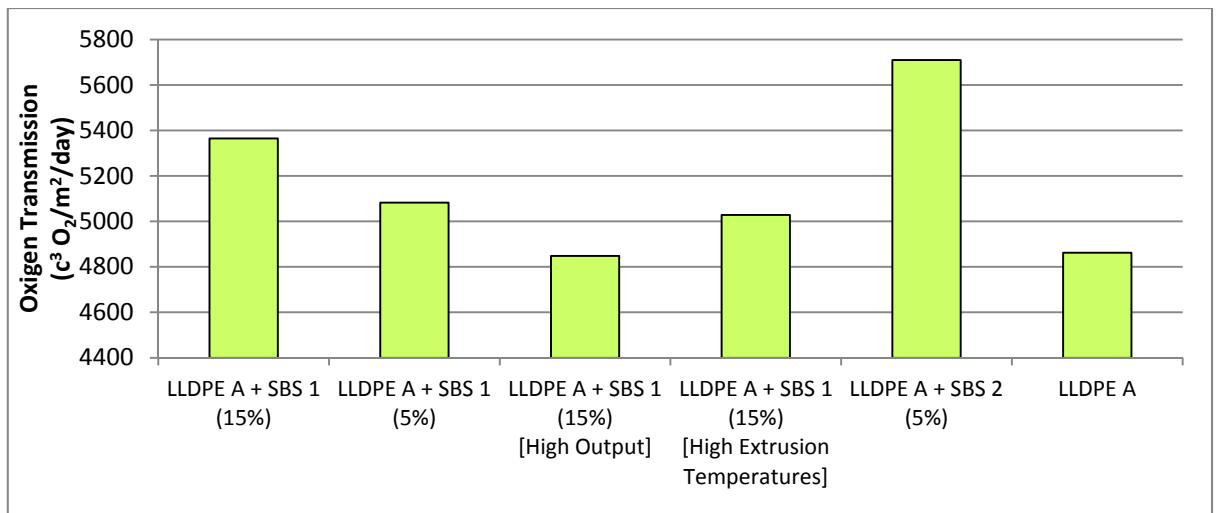
Comparing the blends of 15% of SBS, there is no clear tendency, either. The curves present different maximum force resistance, but the only remarkable observation is that all the blends present the same initiation sealing temperature. and they have the same maximum force resistance.

### 5.2.3.5. Barrier properties

Once all the samples have been tested and the results discussed, the most interesting samples were selected to undergo the Oxygen Transmission test. The chosen samples are those which can present improvements in front of the pure LLDPEs.

In food packaging, oxygen barrier is a very important film property protects the product. It is quantified by the oxygen permeability coefficients which indicate the amount of oxygen that permeates per unit of area and time in a packaging material. Big oxygen permeability coefficients imply lower barrier protection<sup>16</sup>.

<sup>16</sup> <http://www.freepatentsonline.com/EP0236099.pdf>



**Figure 5.29.** Oxygen Transmission test results.

The graph shows that the blend with the higher oxygen barrier is the combination of LLDPE A + SBS 1 extruded with a higher output. At these conditions, the blend improves the barrier in comparison with pure LLDPE. In contrast, the blend of SBS 2 (5%) presents the lower protection barrier.

The results show that SBS 1 decrease the protection barrier in a lower grade. All of these results may be important depending on the application of the blends.

### 5.3. Multilayer blends of LLDPE + SBS

#### 5.3.1. Description

Finally, once the first and second studies are completed, the third study is laid out on the basis of the information obtained from them.

#### Study #3

**Title:** Blends of LLDPE + SBS.

**Description:** Blown multilayer blends of LLDPE + SBS with three layers (A/B/A).

**Purpose:** Study more deeply some selected blends in coextrusion.

As it can be observed in the literature, in connection with the references<sup>[3]</sup>, the purpose was to observe the effect of SBS in a multilayer film. In study #2 some trends were identified that they require verification. In this third study, we selected the monolayer blends for which a more relevant effect of SBS was observed.

The idea is to reproduce those samples in a line which presents the possibility of adding more layers, in this case three, and whose extrusion conditions are more controllable and

reliable. Indeed, the conditions of this line are more similar to the production conditions in a big plant, they are not the same, but the results will be more representative, nearer to the reality. The selected blends and the extrusion conditions are listed and presented in sections 3.1.1 and 3.1.4, respectively.

### 5.3.2. Results & discussion

This section includes the results of the corresponding tests carried out in study #3. The measured properties are the same as in study #2. In the following sections the results of this study are presented, obtained using the same procedures as in previous studies in all tests.

#### 5.3.2.1. Mechanical properties

In the following graphs are represented the results of the mechanical properties which are measured in order to compare the results of the two studies. They are presented in the same order than study #2.

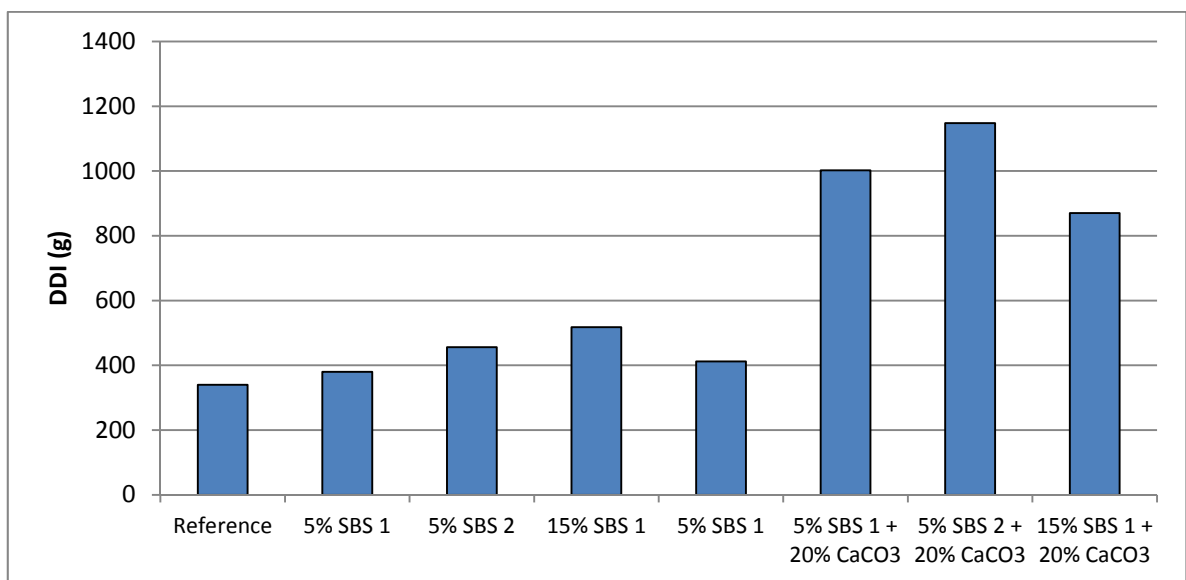
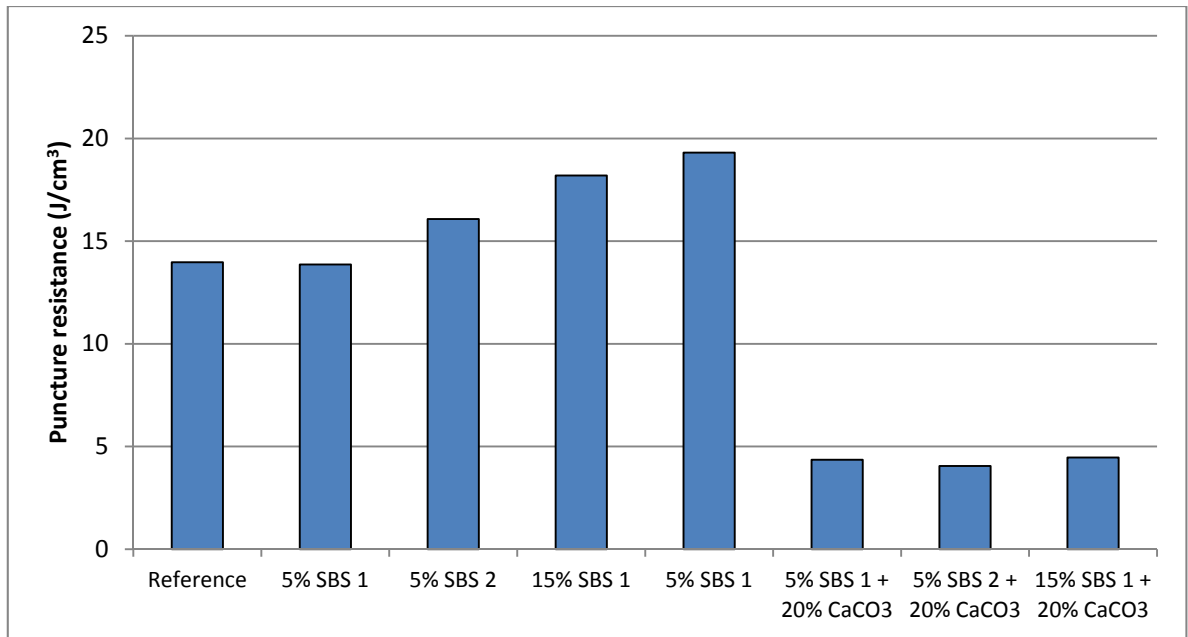


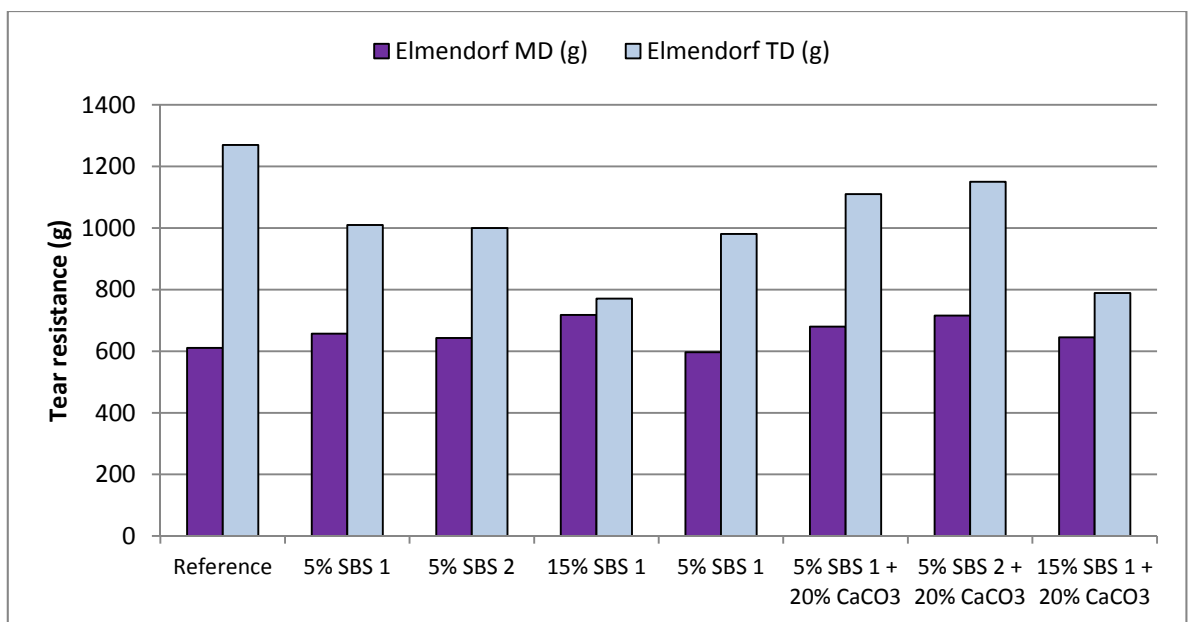
Figure 5.30. Dart Drop Impact test results.

Figure 5.30 shows how the addition of SBS improves the impact resistance of the blends. A 15% of SBS implies better DDI, but it rests analyze the effect in optical properties. However, the addition of CaCO<sub>3</sub> is clearly the most significant effect. However, it is known that Calcium Carbonate implies very bad opticals.



**Figure 5.31.** Puncture test results.

As in DDI, the addition of SBS implies improvements in puncture resistance. However, the addition of Calcium Carbonate reduces it considerably. It is clear that combining SBS with CaCO<sub>3</sub> it is not convenient if the objective is to improve puncture resistance.

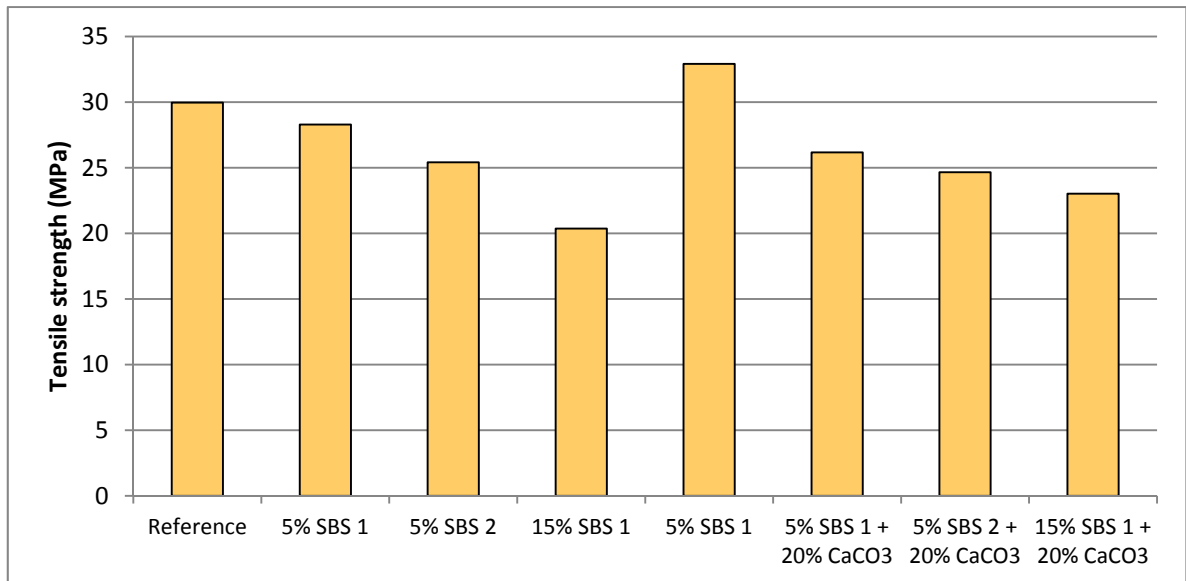


**Figure 5.32.** Elmendorf test results.

Elmendorf test shows how SBS balance the MD/TD tear. The higher the SBS percentage is, the more well-balanced will be the blend. When CaCO<sub>3</sub> is added destabilizes again TD. Therefore, this worsen requires being compare to possible improvements and then decide if it is worth. In general, it seems that there is no difference using SBS 1 or SBS 2.

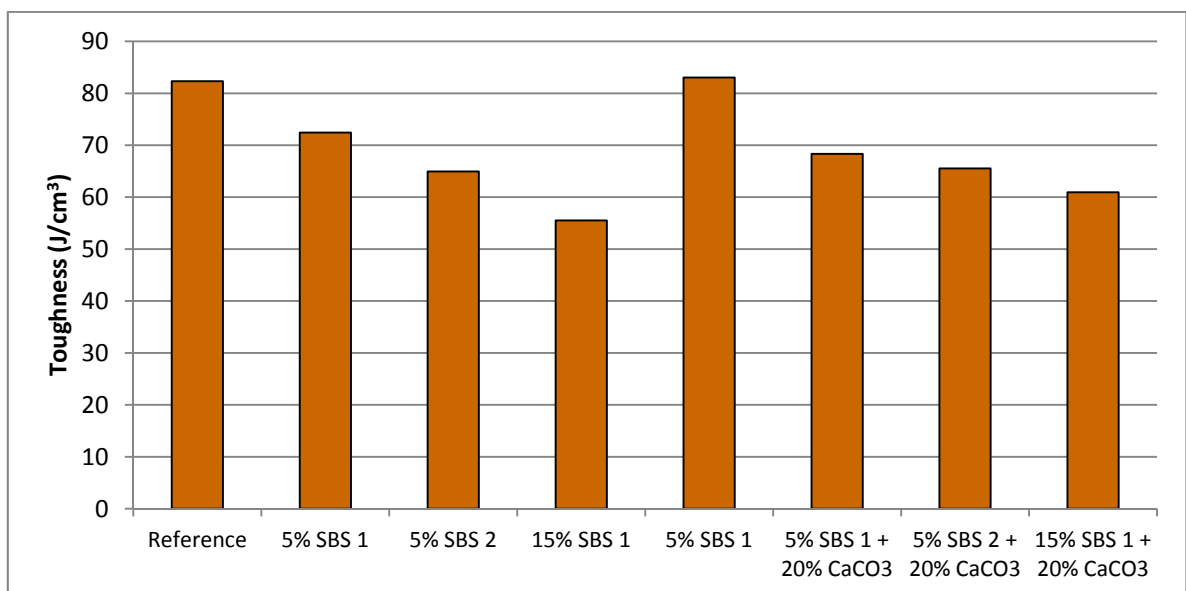
### 5.3.2.2. Elastic and plastic deformations

In the following graphs are presented the behaviour of the blends in terms of deformation.



**Figure 5.33.** Tensile strength results in TD.

In this case, the addition of SBS seems to be negative. Only in one of the duplicated blends appears an improvement. With Calcium Carbonate, the tendency is the same.



**Figure 5.34.** Toughness results in TD.

The tendency is the same explained in figure 5.33, it is exactly the same. Toughness does not seem to be improved by the addition of SBS or both components.

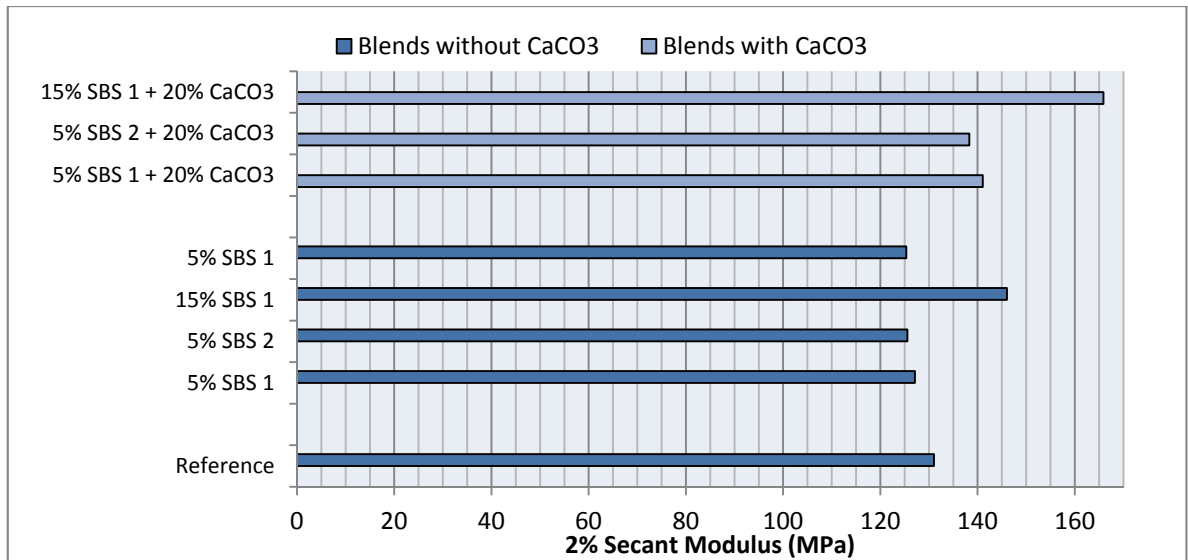


Figure 5.35. Secant Modulus (2%) in TD.

This graph shows the stiffness. The addition of 15% of SBS increases in a small grade the stiffness in comparison with the reference blend. But adding CaCO<sub>3</sub>, the secant modulus, and therefore stiffness, increases even more. Then the blend which presents the best improvement is the last one mentioned, on the top of the graph, because it includes 15% of SBS and 20% of CaCO<sub>3</sub>. However, this combination, until now, it seems not to be the best in other analyzed properties.

**5.3.2.3. Optical properties**

This section includes the results of optical properties. It is well-known that the addition of Calcium Carbonate affects them very negatively. Nevertheless, in some applications, the optics are not the most important thing.

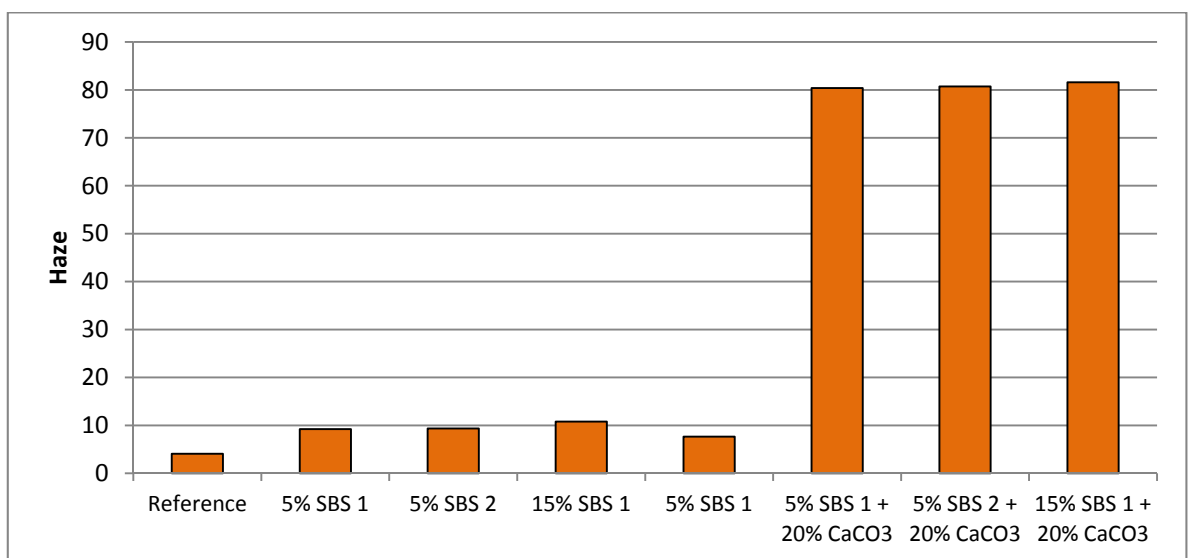
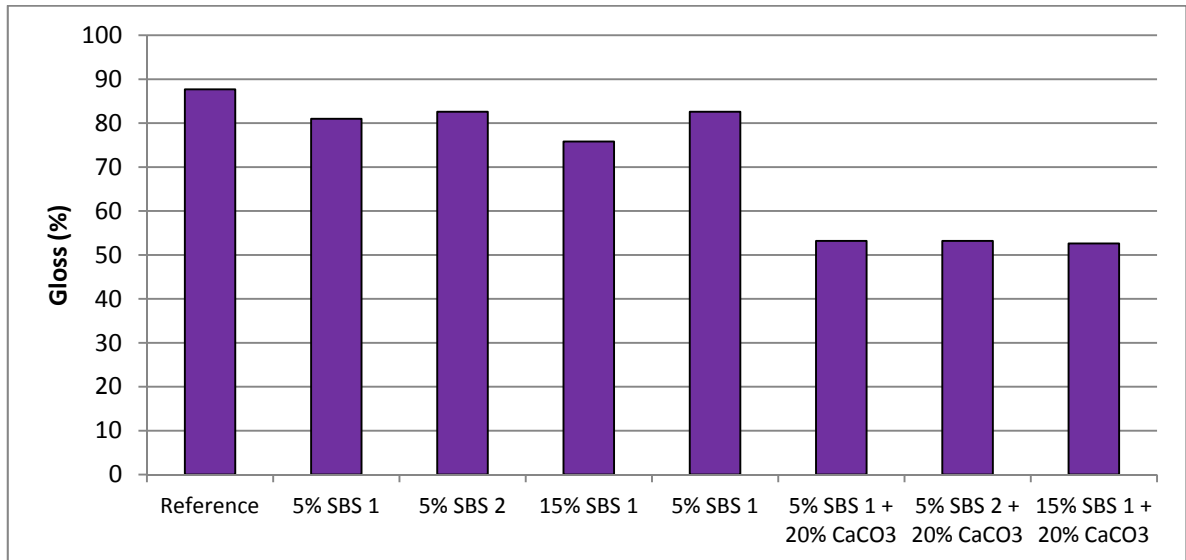


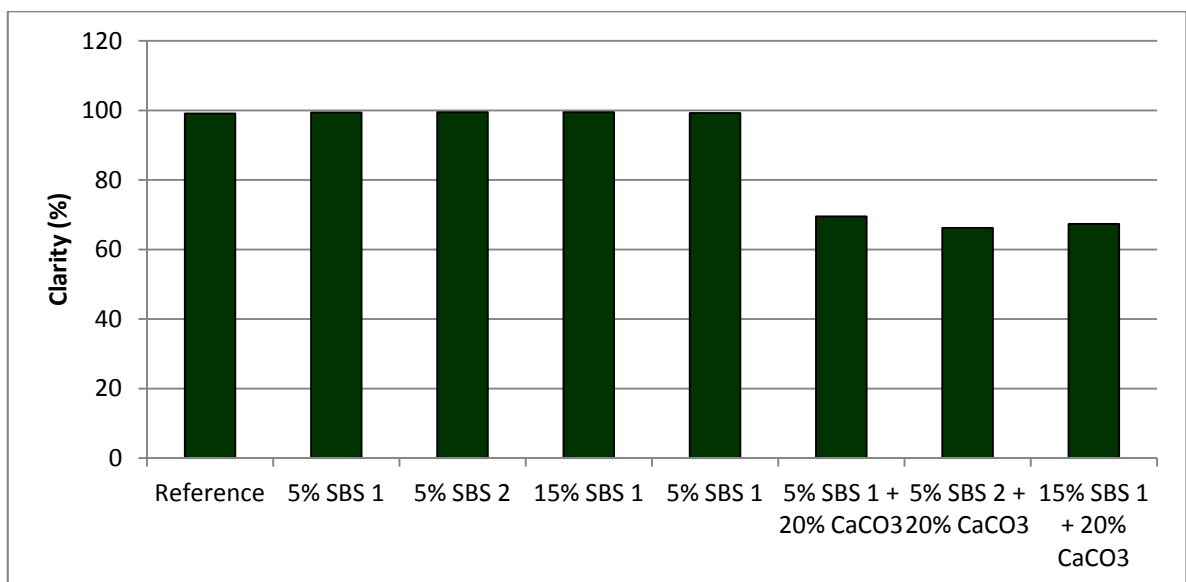
Figure 5.36. Results of Haze.

The addition of SBS implies worsening of haze. Despite the fact, the addition of 5% seems to be acceptable, but not always. One point of haze is a big difference and here the difference is about 3 points at least. Adding Calcium Carbonate the resulting haze is absolutely bad.



**Figure 5.37.** Results of Gloss 45°.

In terms of gloss, SBS worsens it, but the difference is not significant. However, CaCO<sub>3</sub> addition reduces it in a 30%. Probably, the best combination is adding only a 5% of SBS or 15% if a specifically grade of gloss is not required, always guaranteeing a minimum %.

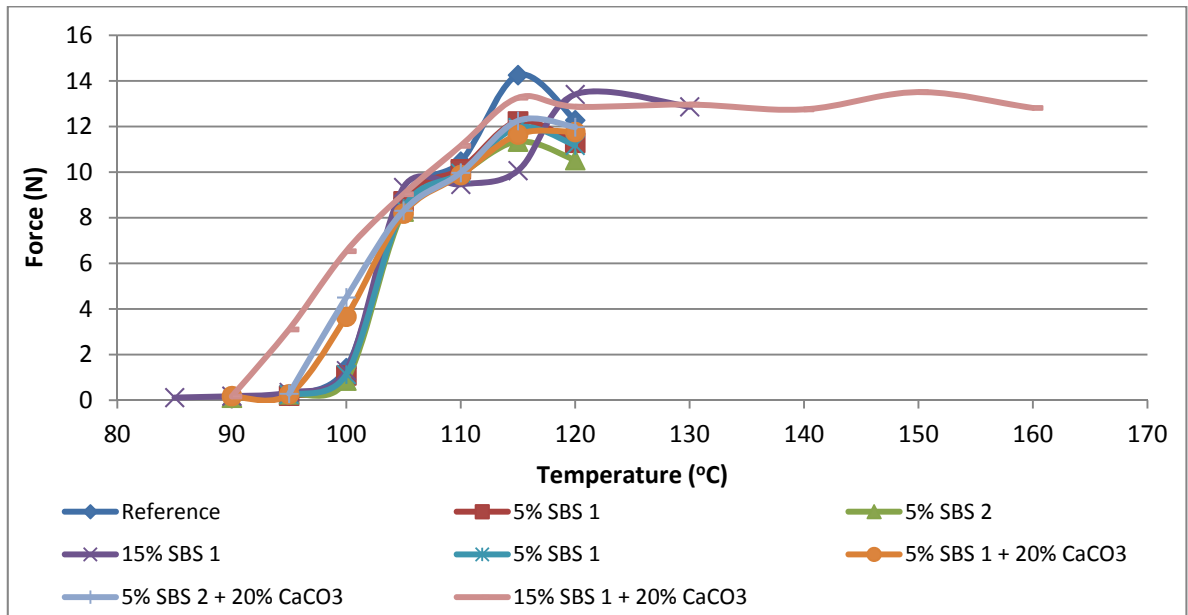


**Figure 5.38.** Results of Clarity.

Clarity results show the same behaviour as haze results. In this case, SBS does not increase significantly the clarity, but Calcium Carbonate decreases in a high grade.

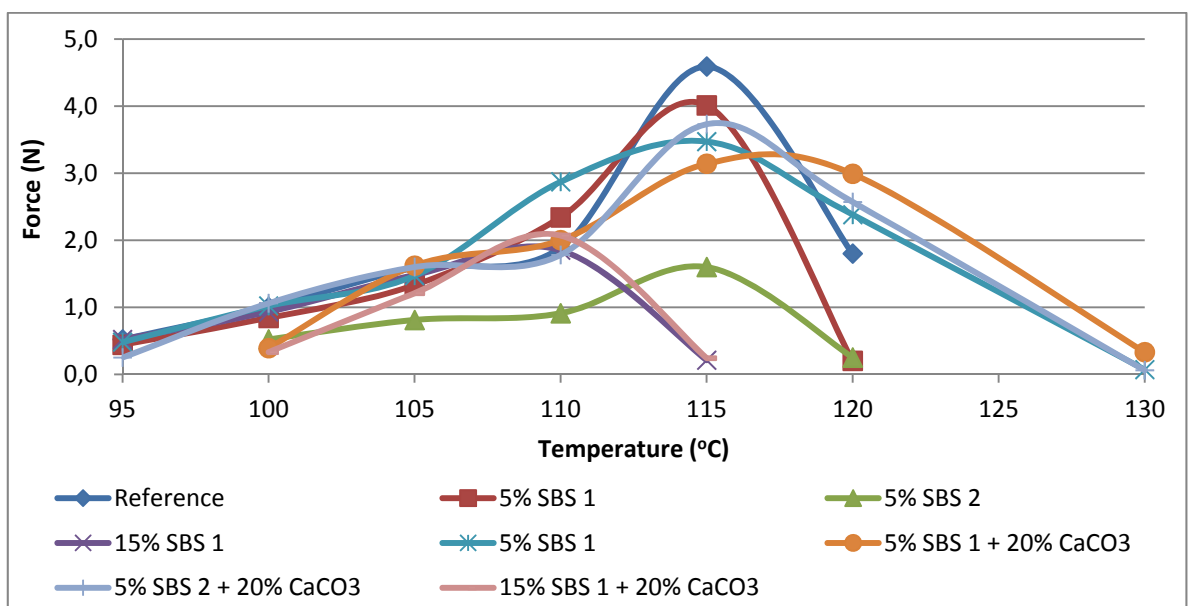
**5.3.2.4. Sealability properties**

Here, the seal strength results are analyzed.



**Figure 5.39.** Seal Strength results.

The combination of 15% of SBS 1 and 20% of CaCO<sub>3</sub> gives high force at higher temperature. The blends with SBS 1 only confirm to some extent what was seen with monolayer films. In this case, pink curve shows a broader seal plateau than the others and, to some extent, the purple curve follows a similar tendency. The difference among them is the presence of CaCO<sub>3</sub>. It seems to intensify the effect, but the more significant effect is the SBS addition because in blends with 5% of SBS, CaCO<sub>3</sub> has no effect.

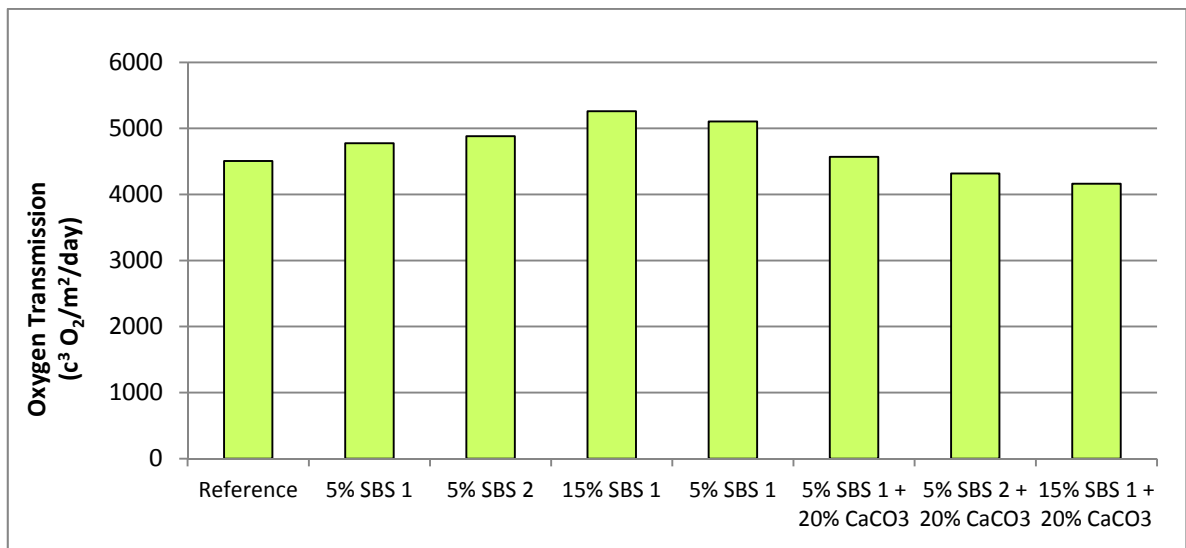


**Figure 5.40.** Hot Tack results of the LLDPE A blends.

The blends of 5% SBS with  $\text{CaCO}_3$  plus one of the blends of 5% SBS 1 present the highest sealing temperature, but they present different initiation sealing temperatures. The graph contains a great deal of variability.

### 5.3.2.5. Barrier properties

Finally, the following graph shows the last test.



**Figure 5.41.** Oxygen Transmission test results.

The blend which presents a higher barrier is the blend with 15% of SBS. The tendency is clear; the addition of SBS implies more oxygen transmission because oxygen barrier blend decreases. But when Calcium Carbonate is added the barrier increase and less oxygen is transmitted. The effect of  $\text{CaCO}_3$  can compensate the reduction in a higher grade. That is, the samples with SBS +  $\text{CaCO}_3$  present worst permeability than reference blend.

### 5.4. Conclusions

Once the results have been discussed, the next step is put in common the conclusions extracted from the second and third studies. The next table summarizes the mentioned information and the idea is compared the conclusion in order to analyse more deeply the behaviour of the studied blends.

**Table 5.10. Studies' results.**

PROPERTIES	STUDY #2	STUDY #3	COMMENTS
<b>Mechanics</b>			
Impact resistance	- For LLDPE A, it stays the same with 5% blends. Otherwise decreases a great deal. - For LLDPE B, it stays the same in all cases.	- SBS increases it, but more with CaCO <sub>3</sub> .	- 5% SBS 1 with LLDPE A stays the same than the pure and with CaCO <sub>3</sub> increase a bit more.
Puncture resistance	- LLDPE A seems adequate with SBS 1. - It is required check 5% blends. - LLDPE B goes down with any blend.	- It tends to increase with SBS. - Add CaCO <sub>3</sub> produces very negative effect.	- SBS increases it. - CaCO <sub>3</sub> is a disaster.
Tear resistance	- MD tear holds or increases, TD tear goes down. - Best balance is maintained for 5% blends.	- SBS balances the MD/TD tear. - Add CaCO <sub>3</sub> increases again the TD.	- Tear ratio (MD/TD) is equalled and orientation, too.
<b>Deformation</b>			
Tensile strength	- Generally no improvement.	- Generally no improvement.	- SBS does not provide improvements.
Toughness	- Generally no improvement.	- Generally no improvement.	
Stiffness	- Only 15% SBS 1 provides significant increase.	- 15% SBS 1 improves and with CaCO <sub>3</sub> even more.	- 15% of SBS 1 increases and CaCO <sub>3</sub> even more.
<b>Optics</b>			
Haze		- The blends with 5% SBS seem to be the only acceptable ones.	- Low % of SBS does not decrease it a lot, but CaCO <sub>3</sub> is unacceptable.
Gloss	- Only blends with 5% of SBS are comparable to pure LLDPE, otherwise much worse.		
Clarity		- CaCO <sub>3</sub> addition highly decreases it.	- CaCO <sub>3</sub> addition highly worsens it.
<b>Sealability</b>			
Seal strength	- LLDPE A blends maybe have a broader seal plateau. - LLDPE B blends do not present significant differences.	- The combination of 15% SBS 1 and CaCO <sub>3</sub> gives high force at higher temperature. - Only blends with SBS 1 have a broader seal plateau.	- Blends with SBS 1 confirm broader seal plateau in LLDPE A blends.
<b>Barrier</b>			
Oxygen transmission	- SBS improves oxygen transmission, CaCO <sub>3</sub> improves permeability.	- SBS may give a bit higher. - CaCO <sub>3</sub> goes lower.	- SBS improves and CaCO <sub>3</sub> worsens.

Therefore, we reach the following conclusions:

- Blends of LLDPE A + SBS 1 present small improvements, in terms of puncture and impact resistance. If we add CaCO<sub>3</sub> increase even more the impact and puncture becomes a disaster.
- MD/TD ratio tends to equal with SBS, but adding CaCO<sub>3</sub> TD increases.
- Tensile strength and toughness do not improve clearly adding SBS.
- Blends of 15% of SBS 1 improve significantly the stiffness and adding CaCO<sub>3</sub> even more.
- Adding a low % of SBS (5%), haze, clarity and gloss do not worsen a lot, but, in terms of haze and gloss, adding CaCO<sub>3</sub> is unacceptable.
- Clarity improves a great deal with the addition of CaCO<sub>3</sub>.
- Blends of LLDPE A + SBS 1 confirm boarder seal plateau.
- SBS tends to increase the oxygen barrier and CaCO<sub>3</sub> to decrease.

It seems clear that LLDPE A offer blends more balance than LLDPE B and SBS 1 tends to present more improvements. Adding small SBS percentages optical and mechanical properties do not worsen a lot and stay the same, respectively. Adding more SBS stiffness and oxygen barrier improve, but the optical and mechanicals can worsen too much.

Therefore, it is true that the addition of SBS to PE does not provide a significantly different performance balance for films, compared to PE only. However some areas such as seal performance should be investigated more in detail. Moreover, it has been studied the addition of CaCO<sub>3</sub> with SBS to PE. It is know that CaCO<sub>3</sub> implies bad results in optical properties, but in some applications can be used a film which does not require a good optics and the combination with SBS and PE could be an interesting option to study, like in terms of oxygen barrier or stiffness.

In food packaging applications, we should give priority to optical properties and permeability to show the products bright and protect them against oxidation.

To sum up, one interesting blend could be the composted by LLDPE A + 5% SBS 1. It seems to be the most interesting or the “most well-balanced” blends with a bit worst optics, but better mechanicals, stiffness, oxygen barrier and broader plateau. Now, as it is mentioned above, it would be interesting to investigate more deeply the behaviour of these blends in terms of seal strength.

## **6. SAFETY IN THE DESIGN OF THE FACILITIES**

### **6.1. Preliminary hazard analysis**

The dangerous scenarios of the process are located in the extruder. The process requires to work at high pressure and temperatures. It works with 3 extruders which imply more variables to control. The following table contains the information so as to improve the control of the process being conscious of the hazards.

Apart from that, we must take into account that polyethylene which is used in big amounts is flammable and can be the cause of possible fires. High dirt accumulation can also originate explosion due to impacts with that dirt. For these reasons, an important point is to always clean the working area to avoid such risks.

Table 6.1. HAZOP.

Junction	Key word	Deviation	Possible causes	Consequences	System reaction	Actions to take
Barrel	LESS	Flow	<ul style="list-style-type: none"> <li>- Level sensor switched off.</li> <li>- No material in the sacks.</li> <li>- Aspirator not running.</li> </ul>	<ul style="list-style-type: none"> <li>- No material, no film.</li> <li>- Burning of the material.</li> <li>- Obstruction, overpressure and possible damage to facilities/personnel.</li> </ul>	<ul style="list-style-type: none"> <li>- Hopper alarm activates.</li> <li>- Panel shows level at 0 mm.</li> </ul>	<ul style="list-style-type: none"> <li>- Install small containers, bigger than 25kg-sacks.</li> <li>- Install light alarms.</li> </ul>
Barrel	MORE	Temperature	<ul style="list-style-type: none"> <li>- Badly-selected temperature profile.</li> <li>- Temperature controller problems.</li> <li>- Temperature (T) sensors not working properly.</li> </ul>	<ul style="list-style-type: none"> <li>- Burning of the material.</li> <li>- No bubble, no film.</li> <li>- Soiling of the die.</li> </ul>	<ul style="list-style-type: none"> <li>- Panels and PC could receive illogical T profile signal and they send them to the controllers.</li> </ul>	<ul style="list-style-type: none"> <li>- Install software to detect possible mistakes in selection of T profile.</li> <li>- Duplicate T sensors.</li> </ul>
Barrel	LESS	Temperature	<ul style="list-style-type: none"> <li>- Badly-selected temperature profile.</li> <li>- Heat losses.</li> <li>- Electrical resistance problem.</li> </ul>	<ul style="list-style-type: none"> <li>- Bad quality film.</li> <li>- Blocked screw.</li> <li>- Screw rupture and possibility of facilities damages.</li> </ul>	<ul style="list-style-type: none"> <li>- Temperature sensors detect problems and send signal to panels and PC which alert the controllers.</li> </ul>	<ul style="list-style-type: none"> <li>- Install temperature sensor at the key points.</li> <li>- Install hot fluid circuit.</li> </ul>
Barrel	MORE	Pressure	<ul style="list-style-type: none"> <li>- Badly-selected pressure.</li> <li>- Obstruction in nozzle and/or hopper.</li> </ul>	<ul style="list-style-type: none"> <li>- Possibility of explosion with damages in the facilities and/or personnel.</li> </ul>	<ul style="list-style-type: none"> <li>- Panel shows critical values in red and sends the signal to the controllers.</li> <li>- Pressure transducer cuts out electricity supply.</li> <li>- At last, higher-pressure rupture disc breaks.</li> </ul>	<ul style="list-style-type: none"> <li>- Install one Rupture Disc per extruder to relieve its pressure.</li> </ul>
Barrel	LESS	Pressure	<ul style="list-style-type: none"> <li>- Screw motor not working properly.</li> <li>- Leaks in the barrel.</li> </ul>	<ul style="list-style-type: none"> <li>- Instabilities in the bubble.</li> <li>- Blocked screw.</li> <li>- Screw rupture and possible damage to facilities/personnel.</li> </ul>	<ul style="list-style-type: none"> <li>- Panel shows drop in pressure in the barrel and sends signal to controllers.</li> </ul>	<ul style="list-style-type: none"> <li>- Check correct functioning of pressure sensors.</li> </ul>

Table 6.1. HAZOP. (Continuation)

Junction	Key word	Deviation	Possible causes	Consequences	System reaction	Actions to take
Barrel	MORE	Amperage	<ul style="list-style-type: none"> <li>- Controller not running correctly.</li> <li>- Accidental rise of tension.</li> </ul>	<ul style="list-style-type: none"> <li>- Pressure and temperature increasing with possibility of explosion with damages to facilities and/or personnel (in case of total obstruction).</li> </ul>	<ul style="list-style-type: none"> <li>- The panel shows the critical values in red and it sends the signal to the controllers.</li> <li>- The pressure transducer cut out the current.</li> <li>- At last, the higher-pressure rupture disc is broken.</li> </ul>	<ul style="list-style-type: none"> <li>- Install another pressure transducer.</li> <li>- Install one Rupture Disc per extruders so as to relieve the extruder pressure.</li> </ul>
Barrel	LESS	Amperage	<ul style="list-style-type: none"> <li>- Electrical problems or tension drops.</li> <li>- Badly-selected set-point.</li> </ul>	<ul style="list-style-type: none"> <li>- Bad quality film.</li> <li>- Blocked screw.</li> <li>- Screw rupture and possible damage to facilities/personnel.</li> </ul>	<ul style="list-style-type: none"> <li>- PC panel shows the values and sends the signal to amperage controller to solve.</li> </ul>	<ul style="list-style-type: none"> <li>- Install extra software to compensate drops and verify set-point regarding material/s.</li> </ul>

## 6.2. Overpressure protection equipments

The extruders are equipped with different protective devices so as to prevent damages to facilities and personnel. The overpressure protective devices are:

- 1) A pressure transducer. It is installed in the flow channel next to the screw, before the breaker plate. It includes an alarm setting for high pressure and a cutout to stop the screw to avoid possible hazard scenarios.
- 2) A High-pressure Rupture Disc (RD). It is included as a further safety option, next to the pressure transducer. It is designed to break at a pressure above the cut-out set-point.
- 3) An Ammeter. The aim of this device is show the drive motor amperage in order to control the running torque on the screw. Detecting tension rises and/or drops is very important.

## 6.3. Personal protection systems

In order to guarantee the safety of the employees it is important to consider the possible risks and choose the appropriate PPE (Personal Protective Equipment). Table 6.2 shows the risks which imply working in the fabrication laboratory with the Collin coextrusion line, the precautions to take and the proper PPE. The objective is risks prevention to work in safe conditions.

**Table 6.2.** Safety key point table.

RISK	PRECAUTION	PPE
Eyes injuries	Use glasses when working in machinery area.	Standard safety glasses.
General injuries (burns, chafing, splash, ...)	Always wear fireproof clothes.	Nomex fireproof clothes (or equivalent).
Damage to skin	Always wear safety shoes.	Standard safety shoes.
Hands injuries	Always use leather gloves, except in tasks in which high touch sensitivity is required or in case another type of gloves is required.	COMETA FLOR-I/TULAN/JUBA leather gloves or equivalent.
Ears injuries	Use ear protectors in noisy areas.	Headphones or ear plugs (Medop-EAR Classic).
Thermal burns	Use heat resistant gloves when manipulating parts at high temperatures or melted polymer.	Kevlar Thermal gloves (number 5805 or equivalent).

**Table 6.2.** Safety key point table. (Continuation)

RISK	PRECAUTION	PPE
Cuts	Use cut resistant gloves when work with cut instruments. When high-sensitivity is required maximize precaution. Use retractile cutter, except in tasks in which you need to apply force during the cut, more cutter length and higher cut precision. In these cases, use NT cutters (or equivalent).	HYFLEX gloves (11-627) or equivalent.
Ozone intoxication	Verify operation of ozone extraction station during corona treatment.	-
Electrocution	Respect prohibition signals. Closet identified with red labels can only be opened and manipulate by the electrical workshop personnel. To open or manipulate electrical panel closets identified with blue labels, a facial protection.	Facial protector (special for electrical works).
Entrapments	Keep hands away of mobile areas in the machine. Do not use loose clothing, ties, scarves, jewelry and/or leave long hair loose near the machines.	-
Musculoskeletal injuries	Do not lift big film rollers single-handed. The same applies to sacks of 25 or more kilograms.	-
Falls	Keep the working area clean of pellets and other objects. Use handrails to go up/down stairs with precaution.	-

Moreover, all the personnel and invited people must satisfy the following general rules obligatorily:

- Do not smoke, except in the smoking areas.
- Do not consume or introduce alcohol or drugs.
- Restricted use of the mobile phone. Consult the areas where it is possible.
- Do not bring or use cameras/video cameras.
- Drive at 30 km/h at most inside the site.
- Do not eat or drink out of the designed places.

Finally, knowing the emergency alarms is indispensable. When the alarm is activated to go to the meeting point and follow the instructions which are specified in the emergency plan.

ATTENTION ALARM	<u>5</u> - 3 - <u>5</u> - 3 - <u>5</u> - 3	seconds
EVACUACION ALARM	<u>½</u> - ½ - <u>½</u> - ½ - <u>½</u> - ½	seconds
ALL-SAFETY ALARM	30	seconds

Note: Every Monday at 12 am an alarm test is performed.

#### 6.4. Protection against dangerous scenarios

The different dangerous scenarios which can occur in the building are the following:

- Chlorine leak.
- Plant emergency.
- Fire in the building.
- Gas leak in Q building (the building next to P).

The following tables contain the basic information about the different scenarios and the instructions to follow when one of them occurs<sup>17</sup>.

**Table 6.3.** Chlorine leak scenario.

<b><u>Chlorine leak</u></b>					
<b>CHLORINE ALARM GOES OFF</b>					
<p>• <b><u>Identify Emergency Command (Security Engineer/Supervisor) and the Security Coordinators</u></b>  <u>Emergency Command</u>: Maintain communication with the Communication Center (CC).  <u>Security coordinators</u>: Check alarm panel of the building and announce over the PA.  <b>“There is a chlorine leak. Keep an eye on the offices and close the windows. Follow the PA instructions.”</b></p>					
<p>• <b><u>Emergency Command or Security Coordinators</u></b>            The CC activates the site emergency plan and announce over the PA:  <b>“Emergency plan has been <u>activated</u>. Close the office door. Go to the nearest confining point. Take the car keys.”</b>  <u>Confining points</u>: P building (DOW room); Q building (LACAT); Temporal huts (DOW room).  <u>Employees/visitors</u>: Take the goggles and masks and go to the confining point with the portable station and check the area sealability.</p>					
<p>• <b><u>Responsible for permits and visitors</u></b>            Manage work permits and inform the visitors accordingly.  <u>Receptionist</u>: Take the in/out panel (previously blocked) and the visitors’ book.</p>					
<b>CONFINING POINT</b>					
<p><u>Security Coordinator</u>: Roll call for both buildings of the department.  <u>Emergency Command or Security Coordinators</u>: Follow the instructions from the CC and transmit them. Check [chloride]. If it is &gt; 1 ppm, check the value with the building next to P.</p>					
<p>• <b><u>Emergency Command or Security Coordinators</u></b></p> <table style="width: 100%; border: none;"> <tr> <td style="width: 50%;"><b><u>EMERGENCY END</u></b></td> <td style="width: 50%;"><b><u>EVACUATION ORDER</u></b></td> </tr> <tr> <td>Activate all-safety alarms and announce: <b>“Emergency end, come back to the work places”</b></td> <td>Turn on evacuation alarm and follow the instructions received from the CC.</td> </tr> </table>		<b><u>EMERGENCY END</u></b>	<b><u>EVACUATION ORDER</u></b>	Activate all-safety alarms and announce: <b>“Emergency end, come back to the work places”</b>	Turn on evacuation alarm and follow the instructions received from the CC.
<b><u>EMERGENCY END</u></b>	<b><u>EVACUATION ORDER</u></b>				
Activate all-safety alarms and announce: <b>“Emergency end, come back to the work places”</b>	Turn on evacuation alarm and follow the instructions received from the CC.				

<sup>17</sup> See section 4.1.3. Plot plan includes the protective measures to minimize the risks (fire blankets, hoses, etc).

**Table 6.4.** Plant emergency scenario.

<b><u>Plant emergency</u></b> <b>THE RECEPTIONIST RED TELEPHONE RINGS</b>	
<b><u>• Receptionist or the nearest person to the telephone</u></b> Take the telephone and listen carefully, identify yourself when CC calls the roll and follow the instructions from the CC. Ask for attention if required.	
<b><u>• Emergency Command or Security Coordinators or Receptionist</u></b> Ask for attention if it is required and announce the instructions from the CC. If the order is to go to meeting point announce: <b>“Take the car keys. Go to the nearest meeting point.”</b>	
<b><u>• Responsible for permits and visitors</u></b> Manage work permits and inform the visitors accordingly. <u>Receptionist</u> : Take the in/out panel (previously blocked) and the visitors’ book.	
<b>MEETING POINT</b>	
<u>Security Coordinator</u> : Call the roll in the both buildings of the department. <u>Emergency Command or Security Coordinators</u> : Check presence of everybody in both buildings and follow the instructions from the CC and transmit them. Ensure that there are enough cars to use in case of evacuation.	
<b><u>• Emergency Command or Security Coordinators</u></b>	
<b><u>EMERGENCY END</u></b>	<b><u>EVACUATION ORDER</u></b>
Active all-safety and announce over the PA: <b>“Emergency end, come back to the work places”</b>	Turn on evacuation alarm and follow the instructions received from the CC.

**Table 6.5.** Fire in the P building scenario.

<b><u>Fire inside of the P building</u></b> <b>SMOKE ALAMR TURNS ON</b>	
<b><u>Person who detects the fire</u></b> Alert the building reception and the CC, take the fire extinguisher and act if it is possible. Call the emergency number, active the emergency alarm and report by PA: <b>“The fire alarm has been turned on. Go to the meeting point following the instructions.”</b>	
<b><u>Security Coordinators</u></b> Check presence of everybody in both buildings.	
<b><u>Emergency Command or Security Coordinators</u></b> Follow the instructions from the CC and transmit them. Ensure that there are enough cars to use in case of evacuation.	
<b><u>Emergency Command or Security Coordinators</u></b>	
<b><u>EMERGENCY END</u></b> Activate all-safety and announce over the PA: <b>“Emergency end, come back to the work places”</b>	<b><u>EVACUATION ORDER</u></b> Turn on evacuation alarm and follow the instructions received from the CC.

## **7. MAINTAINANCE OF FACILITIES**

### **7.1. Cleaning procedure**

Once the samples are manufactured they are brought to the laboratory and the waste is collected and thrown away into waste container. If the study is finished the open sacks are identified and labelled, but if it is a mixture it is usually put in the waste container.

The labelled sacks are driven to the warehouse and the used instruments are put away. Then, the following step is to clean the floor and save the confidential information.

However, if the study did not finish, the step is to close the sacks and put them in a safe place. Then the following steps are the same as before: put the used instruments away, clean the floor and save the confidential information.

### **7.2. Preventive maintenance**

The maintenance is adjusted to the annual program which is carried out with the supplier's equipment. The aim of the following rules is to reduce the external effects which can affect the coextrusion line and guarantee its proper working.

#### a) HANDLE

The resin cannot contain humidity. All the resins must be managed avoiding their contamination by dirt, dust particles, wastes and/or other resins. If it is necessary, material must be heated in an oven to eliminate the humidity.

#### b) STORAGE

The storage of the samples is responsibility of the person who is in charge of the project. The operator or the technicians are not responsible for controlling the state of the samples or their location. However, they can help to improve the safety.

#### c) PACKAGING

The final packaging is responsibility of the person who is in charge of the project.

### **7.3. Predictive maintenance**

The coextrusion line is tested once a year. The Collin professionals test the equipment so as to detect possible evidence of future failures and save time and money. In addition, the following check points should be supervised regularly:

- **Bender, mixer and cooler.** Calibration of all automatic and manual scales.

- **Pumps and fans.** Check the state of this equipment:
  - Detect possible aspiration, system and/or mechanical problems.
  - Check the state of all the parts and their rotation.
  - In case of pumps, detect possible leaks.
  - They can present corrosion problems and cavitation in pumps.
- **Extruders and dies.** The supervision of those parts is very important. It includes:
  - Check the build-up of pellets on the screw hoppers.
  - Clean the drive motor air filters and the vacuum traps.
  - Check the temperature and pressure sensors and controllers.
  - Check the electrical resistances.
  - Analyze the popper screw-barrel alignment and clearance.
  - Check if the counter is working properly.
  - Check the state of the chrome plating on screws and dies.
  - Verify the correct functioning of the screw oil-cooling unit.
- **Downstream.** The following points must be checked:
  - Functioning of water chiller and fans.
  - Possible clog of water spray nozzles.
  - Sizing plugs and the state of the used oil.
  - The state of the blades and their rotation.

## **8. OPERATION MANUAL**

### **8.1. Coextrusion line commissioning**

First of all, we must take into account some safety considerations:

- a) Identify and be near to the emergency buttons.
- b) Pay attention when we manipulate the nip rolls because of entrapment risk.
- c) Check the correct connection of the extruders with the safety circuit.

Then, the instructions are the following:

- 1.** Check the correct working of the water pumps in the extruder's refrigeration circuits and open the inlet and check valves.
- 2.** Connect the main command of each extruder. Press the green button of the extruders.
- 3.** Connect the tension on the panel for the film take-up pressing the green button.
- 4.** Turn on the PC. Go to the *Fecon* program and introduce the user and password. Go to 'process' where you need to introduce the desired temperature profile.
- 5.** Connect the air refrigeration equipments. Establish the working temperature.
- 6.** Check the correct stabilization of the extruders and heads temperatures.
- 7.** Clean the die with a fiber scourer and impregnate it with silicone oil so as to avoid adherences. Remove any degraded polymer remain in the die gap using a metal spatula.
- 8.** Fill the hoppers of LDPE resin using the aspirators next to each extruder.
- 9.** Connect the fans and select a low speed on the film take-up panel.
- 10.** Press the green button to turn on the screws. If the temperature is still low, the screws do not run yet.
- 11.** Introduce a low set-point in the revolution controller (about 5 rpm) until to the polymer leaves the die in a melted state. Controlling the adapter pressure is very important. Never surpass 80 bar and 7 A during the commissioning phase.
- 12.** Let the polymer leaves the die for 5 minutes and then raise the revolutions slightly up to 20/30 rpm. Purge the die for a few minutes.
- 13.** Tie the melted polymer it leaving through the die with the rope or the film which is hanging down from basket. If it is the first time the line is used we can use a rope. The method is to pass the rope through the basket and nip rolls following the course which the film would do. If the line is used before we can stop the machine and cut the film at the height of the roller and we have a piece of film hanging down, using this film as a rope.

14. Control the air flow so as to obtain an approximately 200mm-frost line.
15. Open the bubble swollen-air in order to swell it slowly.
16. Once the polymeric mass has passed through the nip rolls, which are located on the top of the basket, close the nip rolls to push the mass. We can regulate the nip rolls tension to collect the film properly.
17. When the bubble is swollen up to the desired width, close the swollen-air valve.
18. Introduce a low speed in the set-point of the film take-up to raise the film.

## 8.2. Continuous operation procedure

Firstly, we must identify the emergency buttons and then follow these instructions:

1. When the LDPE resin bubble stabilizes, we can fix the adequate temperature profiles for the used resin in the study. The idea is to introduce the new material by doing the changes without running out of any product and to use the existence bubble by modifying a little the parameters when the new material leaves the die.
2. Activating the automatic gravimetric control, we will achieve the needed screws revolutions to ensure a stable production, depending on the number of layers, percentages per layer and specific production of the study samples.
3. Pay attention to the motor consumption and pressure. Never surpass the 7 A and 350 bar.
4. Adjust the temperature and the air flow of the refrigeration systems so as to obtain the desired frost line.
5. If the pressure surpasses the limit or the temperature deviates from the set-point, press the emergency button.
6. The film thickness distribution must be checked when the coextrusion line reaches a steady state, not before. We can modify the thickness distribution playing with screw revolutions (output), the film take-up speed or both.
7. The film diameter or the bent film width is established with the BUR (Blow Up Ratio):

$$BUR = \phi_2 / \phi_1 = \pi \cdot \phi_2 / \pi \cdot \phi_1 = 2L / \pi \cdot \phi_1 \quad (8.1)$$

$$BUR = 0.64L / \phi_1 \quad (8.2)$$

Where  $\phi_1$  is the die diameter,  $\phi_2$  the bubble diameter and L the film width.

8. The bent film can be divided in two sheets using the cutting system and so obtaining two rollers. In contrast, if we do not use this system, we will obtain only one roller. In both cases, the film borders can be cut to improve the thickness distribution.

9. The film collecting axis includes a pneumatic expansion system which maintains cardboard roller fixed. In order to fix it, it must be swollen using the compressed air which had to be removed when we want to take a sample.

10. During the steady state, we will print a pdf copy of the screen panel in order to have the used parameters required to extrude this specific sample.

### **8.3. Coextrusion line stop procedure**

When the samples are finished, the stop procedure is the following:

1. Once the samples are extruded, we need to purge the machine introducing LDPE resin again at process temperature.
2. Purge whole the line until the sample product is eliminated completely. We need to reduce the head temperature slightly up to 180°C.
3. Stop the extruders when they are filled with purge material. Then, reduce the screw revolutions to 0 and press the disconnection button.
4. Load one program for low temperatures and run it for some minutes so as to refrigerate the barrels of the extruders using the fans.
5. Stop the film take-up and press the disconnection button.
6. Press the disconnection buttons of the collecting system.
7. Turn off the cooling ring fan.
8. Disconnect the air of the refrigeration systems.
9. Disconnect the general command of the collecting system.
10. Close the extruders general commands when the barrel temperature decreases to 90°C.
11. Close the water circuit of the extruders.
12. Prepare the line to reheat it the following day. If it is not necessary, stop the PC.
13. Close the air valve for long inactivity periods.
14. Place the protections in the cutting system.

### **8.4. Emergency stop procedure**

The procedure in case of emergency is very simple:

Press one of the emergency buttons and switch off the tension of each extruder. Any stop due to failures in water/air feed has implies that the process is stopped and requires to be restarted manually when the problem has been solved. The plant includes an auxiliary energy system to run the line for short time if it is necessary.

## **9. ECONOMIC EVALUATION**

### **9.1. Project execution budget**

The following tables (9.1, 9.2 and 9.3) show the estimation of the implementation cost of the studied alternatives in order to solve the refrigeration problems. The idea is study which one helps more to reduce time and consumption. If both things are satisfied we have more time to take more costumers.

Each budget presents three costs. In the middle there is the most probable cost, which is used in the cash-flow operations. The estimated cost is the sum of all the equipments<sup>18</sup>:

1. Cooling ring: 20,000\$.
2. IBC: Blower 2,464\$, structure 20,000\$ and structural changes 18,000\$.
3. Refrigeration cycle: Refrigeration cycle 26,500\$ and heat exchanger 4,100\$.

### **9.2. Cash-Flow**

In this section, three different cash-flows are shown. They correspond to the tree suggested alternatives to improve the refrigeration. These cash-flows are different to the typical. This is not a conventional project and the implementation of these alternatives does not imply profits selling the products directly. The manufactured samples are studies. It is a TS&D centre and the idea is: better machine working, more time in our hands and less power and workforce used. However, the employees have a long-term contract. Moreover, with these systems the conditions of the line would be more similar to the industrial conditions and more costumers are interested on test them products before extrude them in a industrial extrusion line. Another improve is the possibility to extrude new products that before we could not do. All of these factors are our incomes. These are the kind of incomes we has TS&D centre, although we could not forget the investment of the company.

The following considerations have been taken into account so as to elaborate the cash-flows (in tables 9.4, 9.5 and 9.6):

- The supposed maintenance cost is 1% of the total investment cost.
- The taxes are the 35% of the total profits.
- The income depends on the chosen alternative.
- The investment in the department is 25,000 € per year.
- The services cost are about 1,200 € per year.

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<sup>18</sup> An operation time 2000 h/year and a Capital Recovery Factor (CRF) of 0,1.



Table 9.2. IBC budget.

ME - Main Equipment	Nº Equipments	2	Estimated Cost	30,518	Cost Index	1.43	Low	Probable	High
<b>CME - Cost Main Equipment</b> Cost Equipment no List P&ID: Very elaborated 2-10%. Preliminary 10-20% CEB - Cost Equipment Basic (without catalyst)									
<b>AC - Average Cost</b>				12,078					
			<b>Comments</b>	<b>Factors</b>					
Installation basic equipment			Medium	11.0%	12.0%	13.0%			
Foundations and structures			Low - Equipments on the ground	0.2%	0.3%	0.4%			
Pipes			Low - Liquids/Solids	10.0%	13.0%	16.0%			
Isolation (equipments and pipes)			Medium - Chemical Plants	13.0%	15.0%	17.0%			
Electricity + Lighting			Liquids	10.0%	11.0%	12.0%			
Instrumentation			Medium	13.0%	15.0%	17.0%			
Unaccounted			Simple process	0.5%	1.0%	1.5%			
Buildings			Indoor equipments	30.0%	40.0%	50.0%			
Others									
<b>Lighting</b>			<b>Building services (%)</b>						
Ventilation and Air conditioning			5%						
Heating			4%						
Plumbing			8%						
Others			3%						
Total services			2%						
Subtotal factors			22%						
Adjustment									
<b>CF - Cost of the Adjust Factors</b>			<b>Low</b>	5%					
			<b>High</b>						
<b>DC - Direct cost of the Plant Limits</b>									
IC - Indirect Cost			29%	of the Direct Cost			40,714	53,043	65,838
Subtotal			14%	of the Subtotal			81,832	98,730	116,094
<b>Unforeseen</b>									
<b>CC - Commissioning Costs</b>									
<b>TC - Total Cost of the Installation</b>									
			(depending on the installation. PE 30 days of charges F + V)				5,000	5,000	5,000
			(max. 4 digits)				<b>110,564</b>	<b>132,362</b>	<b>154,762</b>
			Range				84%	100%	117%

**Table 9.3.** Refrigeration cycle budget.

ME - Main Equipment	Nº Equipments	Estimated Cost	Cost Index	Low	Probable	High
<b>CME - Cost Main Equipment</b>	5		1.43	29,667	32,964	36,260
<b>Cost Equipment no List</b>	P&ID: Very elaborated 2-10%. Preliminary 10-20%		5%	1,483	1,648	1,813
<b>CEB - Cost Equipment Basic (without catalyst)</b>				31,151	34,612	38,073
<b>AC - Average Cost</b>			33,645			
		<b>Comments</b>	<b>Factors</b>			
Installation basic equipment		Medium	7.0%	8.0%	9.0%	
Foundations and structures		Medium - Alloys	1.5%	2.0%	2.5%	
Pipes		Medium - Liquids. Electrolysis.	9.0%	10.0%	11.0%	
Isolation (equipments and pipes)		Medium - Chemical Plants	2.0%	2.5%	3.0%	
Electricity + Lighting		Liquids	3.5%	4.0%	4.5%	
Instrumentation		Medium	2.5%	3.0%	4.5%	
Unaccounted		Complex process	4.0%	5.0%	6.0%	
Buildings		Outdoor equipments	1.5%	2.5%	3.5%	
Others						
	<b>Building services (%)</b>					
Lighting	5%					
Ventilation and Air conditioning	4%					
Heating	8%					
Plumbing	3%					
Others	2%					
Total services	22%					
Subtotal factors			0.3%	1%	1%	
Adjustment			31%	38%	45%	
	<b>Low</b>					
	5%					
	<b>High</b>					
	-5%					
<b>CF - Cost of the Adjust Factors</b>			33%	38%	43%	10,247
<b>DC - Direct cost of the Plant Limits</b>						12,997
<b>IC - Indirect Cost</b>	29%	of the Direct Cost				16,193
Subtotal						41,398
<b>Unforeseen</b>	14%	of the Subtotal				54,266
<b>CC - Commissioning Costs</b>		(depending on the installation. PE 30 days of charges F + V)				12,005
<b>TC - Total Cost of the Installation</b>		(max. 4 digits)				13,806
		Range				53,404
						7,476
						3,200
						3,200
						56,604
						64,615
						88%
						100%
						73,203
						113%

Table 9.4. Cash-flow of the refrigeration cycle implementation.

Year	2012	2013	2014	2015	2016	2017	2018	2019	2020	2021
<b>Investment (Fix Capital)</b>	Refrigeration cycle	64,615								
<b>Incomes</b>	TS&D Capital	25,000	25,250	25,503	25,758	26,015	26,538	26,803	27,071	27,342
	New costumers	12,000	13,200	14,520	15,972	17,569	21,259	23,385	25,723	28,295
	New products	8,000	8,800	9,680	10,648	11,713	14,172	15,590	17,149	18,864
		45,000	47,250	49,703	52,378	55,297	61,969	65,778	69,943	74,501
<b>Variable costs</b>	Operation costs	2,312	2,312	2,312	2,312	2,312	2,312	2,312	2,312	2,312
	Raw Materials	25,000	25,250	25,503	25,758	26,015	26,538	26,803	27,071	27,342
	Utilities	10,000	10,100	10,201	10,303	10,406	10,615	10,721	10,829	10,937
		37,312	37,662	38,016	38,373	38,733	39,465	39,837	40,212	40,591
<b>Fix costs</b>	Services cost	1,200	1,200	1,200	1,200	1,200	1,200	1,200	1,200	1,200
	Maintenance cost	646	646	646	646	646	646	646	646	646
		1,846	1,846	1,846	1,846	1,846	1,846	1,846	1,846	1,846
<b>PBT</b>	Profits Before Taxes	5,842	7,742	9,841	12,159	14,718	20,658	24,095	27,885	32,064
<b>Amortization</b>	Refrigeration cycle	6,462	6,462	6,462	6,462	6,462	6,462	6,462	6,462	6,462
<b>Taxes</b>	35%	2,045	2,710	3,444	4,256	5,151	6,140	7,230	8,433	9,760
<b>Cash-Flow</b>	Profits After Taxes	-60,818	5,032	6,397	7,903	9,567	11,402	15,662	18,125	20,842
<b>Cash-Flow</b>	Accumulated	-60,818	-55,786	-49,389	-41,486	-31,919	-20,517	-7,089	8,572	26,698
<b>C-Flow Discounted</b>	1,00%	-60,818	5,032	6,333	7,748	9,285	10,957	12,776	14,754	16,906
	3,00%	-60,818	5,032	6,210	7,450	8,755	10,131	11,583	13,116	14,738
	4,00%	-60,818	5,032	6,151	7,307	8,505	9,747	11,037	12,378	13,774
	6,00%	-60,818	5,032	6,034	7,034	8,032	9,032	10,034	11,041	12,054
										13,076

Table 9.5. Cash-flow of the IBC implementation.

Year	2012	2013	2014	2015	2016	2017	2018	2019	2020	2021
<b>Investment (Fix Capital) IBC</b>	132,362									
<b>Incomes</b>										
TS&D Capital	25,000	25,250	25,503	25,758	26,015	26,275	26,538	26,803	27,071	27,342
New costumers	20,000	22,000	24,200	26,620	29,282	32,210	35,431	38,974	42,872	47,159
New products	15,000	16,500	18,150	19,965	21,962	24,158	26,573	29,231	32,154	35,369
	60,000	63,750	67,853	72,343	77,259	82,643	88,543	95,008	102,097	109,870
<b>Variable costs</b>										
Operation costs	2,186	1,312	1,202	1,093	1,093	1,093	1,093	1,093	1,093	1,093
Raw Materials	30,000	30,300	30,603	30,909	31,218	31,530	31,846	32,164	32,486	32,811
Utilities	15,000	15,150	15,302	15,455	15,609	15,765	15,923	16,082	16,243	16,405
	47,186	46,762	47,107	47,456	47,920	48,388	48,861	49,339	49,821	50,309
<b>Fix costs</b>										
Services cost	2,000	2,000	2,000	2,000	2,000	2,000	2,000	2,000	2,000	2,000
Maintenance cost	1,324	1,324	1,324	1,324	1,324	1,324	1,324	1,324	1,324	1,324
	3,324	3,324	3,324	3,324	3,324	3,324	3,324	3,324	3,324	3,324
<b>PBT</b>	9,491	13,665	17,422	21,562	26,015	30,931	36,358	42,346	48,952	56,238
<b>Amortization</b>	13,236	13,236	13,236	13,236	13,236	13,236	13,236	13,236	13,236	13,236
<b>Taxes</b>	3,322	4,783	6,098	7,547	9,105	10,826	12,725	14,821	17,133	19,683
<b>Cash-Flow</b>										
Profits After Taxes	-126,194	8,882	11,324	14,016	16,910	20,105	23,632	27,525	31,819	36,555
<b>Cash-Flow</b>										
Accumulated	-126,194	-117,311	-105,987	-91,971	-75,062	-54,956	-31,324	-3,799	28,020	64,574
<b>C-Flow Discounted</b>										
1,00%	-126,194	8,882	11,212	13,739	16,412	19,321	22,486	25,930	29,678	33,758
3,00%	-126,194	8,882	10,995	13,211	15,475	17,863	20,386	23,052	25,872	28,857
4,00%	-126,194	8,882	10,889	12,958	15,033	17,186	19,424	21,753	24,180	26,710
6,00%	-126,194	8,882	10,683	12,474	14,198	15,925	17,660	19,404	21,161	22,935

Table 9.4. Cash-flow of the cooling ring implementation.

Year	2012	2013	2014	2015	2016	2017	2018	2019	2020	2021
<b>Investment (Fix Capital)</b>	Cooling ring									
	56,067									
<b>Incomes</b>										
TS&D Capital	25,000	25,250	25,503	25,758	26,015	26,275	26,538	26,803	27,071	27,342
New costumers	12,000	13,200	14,520	15,972	17,569	19,326	21,259	23,385	25,723	28,295
New products	6,000	6,600	7,260	7,986	8,785	9,663	10,629	11,692	12,862	14,148
	43,000	45,050	47,283	49,716	52,369	55,264	58,426	61,880	65,656	69,785
<b>Variable costs</b>										
Operation costs	1,207	1,086	1,026	1,026	1,026	1,026	1,026	1,026	1,026	1,026
Raw Materials	25,000	25,250	25,503	25,758	26,015	26,275	26,538	26,803	27,071	27,342
Utilities	10,000	10,100	10,201	10,303	10,406	10,510	10,615	10,721	10,829	10,937
	36,207	36,436	36,729	37,086	37,447	37,811	38,179	38,550	38,926	39,305
<b>Fix costs</b>										
Services cost	1,200	1,200	1,200	1,200	1,200	1,200	1,200	1,200	1,200	1,200
Maintenance cost	561	561	561	561	561	561	561	561	561	561
	1,761	1,761	1,761	1,761	1,761	1,761	1,761	1,761	1,761	1,761
<b>PBT</b>	5,033	6,853	8,793	10,869	13,161	15,693	18,487	21,569	24,970	28,720
<b>Amortization</b>	5,607	5,607	5,607	5,607	5,607	5,607	5,607	5,607	5,607	5,607
<b>Taxes</b>	35%	2,399	3,077	3,804	4,606	5,492	6,470	7,549	8,739	10,052
<b>Cash-Flow</b>										
Profits After Taxes	-52,796	4,455	5,715	7,065	8,555	10,200	12,016	14,020	16,230	18,668
<b>Cash-Flow</b>										
Accumulated	-52,796	-48,341	-42,626	-35,561	-27,006	-16,806	-4,790	9,230	25,461	44,128
<b>C-Flow Discounted</b>										
1,00%	-52,796	4,455	5,659	6,925	8,303	9,802	11,433	13,207	15,138	17,239
3,00%	-52,796	4,455	5,549	6,659	7,829	9,063	10,365	11,741	13,197	14,737
4,00%	-52,796	4,455	5,495	6,532	7,605	8,719	9,876	11,080	12,334	13,640
6,00%	-52,796	4,455	5,392	6,287	7,183	8,080	8,979	9,884	10,794	11,712

### 9.3. Investment global evaluation

The different alternatives have been evaluated in order to know if their implementation would be profitable or not. The following tables contain the results:

**Table 9.5.** Refrigeration cycle profitability evaluation.

Interest	1.0%	2.0%	4.0%	6.0%	8.0%	10.0%	12.0%	14.0%	16.0%
<b>VAN (€)</b>	40,792	34,650	23,953	15,044	7,602	1,369	-3,863	-8,265	-11,973
<b>TIR (%)</b>	10.49								
<b>Payback</b>	6.5 years								

**Table 9.6.** Internal Bubble Cooling profitability evaluation.

Interest	1.0%	2.0%	4.0%	6.0%	8.0%	10.0%	12.0%	14.0%	16.0%
<b>VAN (€)</b>	52,899	42,284	23,829	8,505	-4,252	-14,896	-23,793	-31,239	-37,477
<b>TIR (%)</b>	7.29								
<b>Payback</b>	7.25 years								

**Table 9.7.** Cooling ring profitability evaluation.

Interest	1.0%	2.0%	4.0%	6.0%	8.0%	10.0%	12.0%	14.0%	16.0%
<b>VAN (€)</b>	38,074	32,561	22,956	14,954	8,266	2,662	-2,047	-6,010	-9,352
<b>TIR (%)</b>	11.09								
<b>Payback</b>	6.5 years								

The reference interest (i) is 6%. All alternatives are profitable because they present positive VAN and the TIR is bigger than 6%. Positive VAN and TIR > i means that invest this capital in that implementation is better than invest at 6% of interest. Observing the tables 9.5 – 9.7 we can appreciate that the lowest VAN is the IBC VAN. This system is the most expensive and now is not available, but at the same time is the best option in terms of improvement and development. Another important factor is the payback which indicates the required time to recover the investment, in this case 6-7 years, probably a bit too slow.

Implementing an IBC system we increase the bubble stability around 40-50% and consequently the velocity of the process. However, the highest output we can achieve with a 60 mm die is 22.5 kg/h. Higher values are not possible with this width.

In conclusion, the economic evaluation shows that a good idea would be implement a new cooling ring or install a refrigeration cycle and pay attention to the market in the development of IBC system for the used coextrusion line because is the implementation which really improve significantly the refrigeration of the line and should offer a better successful percentage in the new products which the plant develop.


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## A.1. ANNEXES

### A.1.1. Instrumentation


**Table A.1.** Level sensor datasheet.

<b>UNIVERITAT ROVIRA I VIRGILI</b> CHEMICAL ENGINEERING DEPARTMENT Escola Tècnica Superior d'Enginyeria Química	<b>Instrument ID:</b> LT	
<u>GENERAL INFORMATION</u>		
<b>Type</b>	Level sensor	
<b>Supplier</b>	Radar	
<b>Model</b>	SICK DT20-P130B1000	
<u>CHARACTERISTICS</u>		
<b>Measuring range</b>	90 to 1000 mm	
<b>Precision</b>	±1.5 mm	
<b>Material</b>	Rugged. metal housing	
<u>UNITS</u>	3	


**Table A.2.** Alarm buzzer datasheet..

<b>UNIVERITAT ROVIRA I VIRGILI</b> CHEMICAL ENGINEERING DEPARTMENT Escola Tècnica Superior d'Enginyeria Química	<b>Instrument ID:</b> AL	
<u>GENERAL INFORMATION</u>		
<b>Type</b>	Alarm buzzer	
<b>Supplier</b>	Audible	
<b>Model</b>	SWITCHES PLUS 55-04301	
<u>CHARACTERISTICS</u>		
<b>Frequency</b>	60 Hz	
<b>Power consumption</b>	13 mA	
<b>Material</b>	Pastic and metal	
<u>UNITS</u>	3	


**Table A.3.** Flow indicator sensor datasheet..

<b>UNIVERITAT ROVIRA I VIRGILI</b> CHEMICAL ENGINEERING DEPARTMENT Escola Tècnica Superior d'Enginyeria Química	<b>ID:</b> FIT	
<u>GENERAL INFORMATION</u>		
<b>Type</b>	Flow indicator sensor	
<b>Supplier</b>	Ultrasounds	
<b>Model</b>	SICK FFUS15-1G1/O	
<u>CHARACTERISTICS</u>		
<b>Flow range</b>	0.9 to 36 L/min	
<b>Precision</b>	0.006 L/min	
<b>Material</b>	High-quality polysulfone	
<u>UNITS</u>	7	


**Table A.4.** Ball valve datasheet..

<b>UNIVERITAT ROVIRA I VIRGILI</b> CHEMICAL ENGINEERING DEPARTMENT Escola Tècnica Superior d'Enginyeria Química	<b>Instrument ID:</b> VLV	
<u>GENERAL INFORMATION</u>	Ball valve	
<b>Type</b>	High pressure	
<b>Supplier</b>	TECVAL	
<b>Model</b>	VB-12	
<u>CHARACTERISTICS</u>		
<b>Temperature rang</b>	-20 to 200°C	
<b>Pressure</b>	Up to 210 bar	
<b>Material</b>	Stainless steel AISI-316	
<u>UNITS</u>	4	


**Table A.5.** Check valve datasheet..

<b>UNIVERITAT ROVIRA I VIRGILI</b> CHEMICAL ENGINEERING DEPARTMENT Escola Tècnica Superior d'Enginyeria Química	<b>Instrument ID:</b> CHK	
<u>GENERAL INFORMATION</u>	Check valve	
<b>Type</b>	Poppet check	
<b>Supplier</b>	TECVAL	
<b>Model</b>	VR-01	
<u>CHARACTERISTICS</u>		
<b>Temperature rang</b>	-20 to 400°C	
<b>Pressure</b>	Up to 210 bar	
<b>Material</b>	Stainless steel AISI-316	
<u>UNITS</u>	3	


**Table A.6.** Temperature sensor datasheet..

<b>UNIVERITAT ROVIRA I VIRGILI</b> CHEMICAL ENGINEERING DEPARTMENT Escola Tècnica Superior d'Enginyeria Química	<b>Instrument ID:</b> TT	
<u>GENERAL INFORMATION</u>	Temperature sensor	
<b>Type</b>	Thermocouple	
<b>Supplier</b>	GUEMISSA	
<b>Model</b>	Pt100-STSN	
<u>CHARACTERISTICS</u>		
<b>Temperature rang</b>	100 to 1200°C	
<b>Precision</b>	±0.1°C	
<b>Material</b>	Stainless steel AISI-316 PVC (Head)	
<u>UNITS</u>	21	


**Table A.7.** Pressure transducer datasheet..

<b>UNIVERITAT ROVIRA I VIRGILI</b> CHEMICAL ENGINEERING DEPARTMENT Escola Tècnica Superior d'Enginyeria Química	<b>Instrument ID:</b> PT	
<u>GENERAL INFORMATION</u>	Pressure transducer	
<b>Type</b>	Differential	
<b>Supplier</b>	HONEYWELL	
<b>Model</b>	FP-2000	
<u>CHARACTERISTICS</u>	0.04 to 690 bar	
<b>Pressure range</b>	±0.1% (50 mV)	
<b>Accuracy</b>	Ha C276 & 316L stainless steel	
<b>Material</b>		
<u>UNITS</u>	12	


**Table A.8.** Ratemeter datasheet..

<b>UNIVERITAT ROVIRA I VIRGILI</b> CHEMICAL ENGINEERING DEPARTMENT Escola Tècnica Superior d'Enginyeria Química	<b>Instrument ID:</b> VT	
<u>GENERAL INFORMATION</u>	Ratemeter	
<b>Type</b>	Board	
<b>Supplier</b>	SIMEX	
<b>Model</b>	STI-94	
<u>CHARACTERISTICS</u>	1.5 to 50000 rpm	
<b>Rotational speed</b>	±0.02%	
<b>Precision</b>	NORYL – GFN2S E1	
<b>Material</b>		
<u>UNITS</u>	3	


**Table A.9.** Wireless temperature sensor datasheet..

<b>UNIVERITAT ROVIRA I VIRGILI</b> CHEMICAL ENGINEERING DEPARTMENT Escola Tècnica Superior d'Enginyeria Química	<b>Instrument ID:</b> TT	
<u>GENERAL INFORMATION</u>	Temperature sensor	
<b>Type</b>	Wireless sensor	
<b>Supplier</b>	BEANAIR	
<b>Model</b>	V1R0	
<u>CHARACTERISTICS</u>	-70 to 380°C	
<b>Temperature rang</b>	±0.5°C	
<b>Precision</b>	Stainless steel and plastic	
<b>Material</b>		
<u>UNITS</u>	2	


**Table A.10.** Distance sensor datasheet..

<b>UNIVERITAT ROVIRA I VIRGILI</b> CHEMICAL ENGINEERING DEPARTMENT Escola Tècnica Superior d'Enginyeria Química	<b>Instrument ID:</b> SF	
<u>GENERAL INFORMATION</u>	Distance sensor	
<b>Type</b>	Photoelectric	
<b>Supplier</b>	IFM	
<b>Model</b>	OGHLFPKG	
<u>CHARACTERISTICS</u>	20 to 200 mm	
<b>Measuring rang</b>	1.5 mm	
<b>Precision</b>	PMMA (Lent)	
<b>Material</b>	Stainless steel 316L (cover)	
<u>UNITS</u>	2	

**Table A.11.** Pressure indicator datasheet..

<b>UNIVERITAT ROVIRA I VIRGILI</b> CHEMICAL ENGINEERING DEPARTMENT Escola Tècnica Superior d'Enginyeria Química	<b>Instrument ID:</b> PT	
<u>GENERAL INFORMATION</u>	Pressure indicator	
<b>Type</b>	Bourdon-manometer	
<b>Supplier</b>	MARSH	
<b>Model</b>	J0472	
<u>CHARACTERISTICS</u>	0 to 350 bar	
<b>Temperature rang</b>	±1 bar	
<b>Precision</b>	General steel	
<b>Material</b>		
<u>UNITS</u>	7	

**Table A.12.** Ball valve datasheet.

<b>UNIVERITAT ROVIRA I VIRGILI</b> CHEMICAL ENGINEERING DEPARTMENT Escola Tècnica Superior d'Enginyeria Química	<b>Instrument ID:</b> VLV	
<u>GENERAL INFORMATION</u>	Ball valve	
<b>Type</b>	Miniature	
<b>Supplier</b>	TECVAL	
<b>Model</b>	VB-08	
<u>CHARACTERISTICS</u>	-20 to 200°C	
<b>Pressure rang</b>	Up to 210 bar	
<b>Precision</b>	Stainless steel AISI-316	
<b>Material</b>		
<u>UNITS</u>	3	

**A.1.2. Refrigeration cycle****"ID number: 112121"****"Title: Assessment of blends of SBS copolymer with polyethylene for film applications"****"Student: Joan Ventura Fàbregas"****"Refrigerator cycle"**

P[0]=1,01  
 T[0]=298  
 h[0]=Enthalpy(R134a;P=P[0];T=T[0])  
 s[0]=Entropy(R134a;P=P[0];T=T[0])  
  
 P[1]=22  
 x[1]=1  
 h[1]=Enthalpy(R134a;P=P[1];x=x[1])  
 s[1]=Entropy(R134a;P=P[1];x=x[1])  
 T[1]=Temperature(R134a;P=P[1];x=x[1])  
 v[1]=Volume(R134a;P=P[1];x=x[1])  
 epsilon[1]=(h[1]-h[0])-T[0]\*(s[1]-s[0])  
  
 P[2]=27  
 ss2=s[1]  
 h2s=Enthalpy(R134a;P=P[2];s=ss2)  
 eta\_is=0,7  
 eta\_is=(h2s-h[1])/(h[2]-h[1])  
  
 s[2]=Entropy(R134a;P=P[2];h=h[2])  
 T[2]=Temperature(R134a;P=P[2];h=h[2])  
 x[2]=Quality(R134a;P=P[2];h=h[2])  
 v[2]=Volume(R134a;P=P[2];h=h[2])  
 epsilon[2]=(h[2]-h[0])-T[0]\*(s[2]-s[0])  
 Wcomp=m\_dot\_23\*(h[2]-h[1])  
  
 P[3]=P[2]  
 T[3]=263  
 h[3]=Enthalpy(R134a;P=P[3];T=T[3])  
 s[3]=Entropy(R134a;P=P[3];T=T[3])  
 x[3]=Quality(R134a;P=P[3];T=T[3])  
 v[3]=Volume(R134a;P=P[3];T=T[3])  
 epsilon[3]=(h[3]-h[0])-T[0]\*(s[3]-s[0])  
 Wvalv=m\_dot\_23\*(h[3]-h[4])  
  
 P[4]=P[1]  
 h[4]=h[3]  
 T[4]=Temperature(R134a;P=P[4];h=h[4])  
 s[4]=Entropy(R134a;P=P[4];h=h[4])  
 x[4]=Quality(R134a;P=P[4];h=h[4])  
 v[4]=Volume(R134a;P=P[4];h=h[4])  
 epsilon[4]=(h[4]-h[0])-T[0]\*(s[4]-s[0])  
  
**"Air condenser"**  
 P[6]=1  
 T[6]=298  
 cp[6]=Cp(Air;T=T[6])  
 h[6]=Enthalpy(Air;T=T[6])  
 s[6]=Entropy(Air;P=P[6];T=T[6])  
 epsilon[6]=(h[6]-h[0])-T[0]\*(s[6]-s[0])  
 m\_dot\_23=0,1  
 Q23=m\_dot\_23\*(h[3]-h[2])  
 Q67=-Q23  
 T[7]=313  
 Q67=m\_dot\_67\*cp[6]\*(T[7]-T[6])

**"Water process"**

P[8]=1  
 T[8]=288  
 cp[8]=Cp(Water;P=P[8];T=T[8])  
 h[8]=Enthalpy(Water;P=P[8];T=T[8])  
 s[8]=Entropy(Water;P=P[8];T=T[8])  
 epsilon[8]=(h[8]-h[0])-T[0]\*(s[8]-s[0])  
  
 m\_dot\_41=m\_dot\_23  
 Q41=m\_dot\_41\*(h[1]-h[4])  
 Q89=-Q41  
 T[9]=279  
 Q89=m\_dot\_89\*cp[8]\*(T[9]-T[8])

**"Pre-cooled air"**

T[11]=298  
 T[12]=284  
 m\_dot\_1112=0,22  
  
 Q\_1112=m\_dot\_1112\*cp[6]\*(T[12]-T[11])  
 Q\_910=-Q\_1112  
 m\_dot\_910=m\_dot\_89  
 Q\_910=m\_dot\_910\*cp[8]\*(T[10]-T[9])

**"Graph points"**

P[5]=P[1]  
 T[5]=T[1]  
 h[5]=h[1]  
 s[5]=s[1]  
 v[5]=v[1]

**"Temperatures"**

Ti[0]=T[0]-273  
 Ti[1]=T[1]-273  
 Ti[2]=T[2]-273  
 Ti[3]=T[3]-273  
 Ti[4]=T[4]-273  
 Ti[5]=T[5]-273  
 Ti[6]=T[6]-273  
 Ti[7]=T[7]-273  
 Ti[8]=T[8]-273  
 Ti[9]=T[9]-273  
 Ti[10]=T[10]-273  
 Ti[11]=T[11]-273  
 Ti[12]=T[12]-273

**"Variables (Units)"**

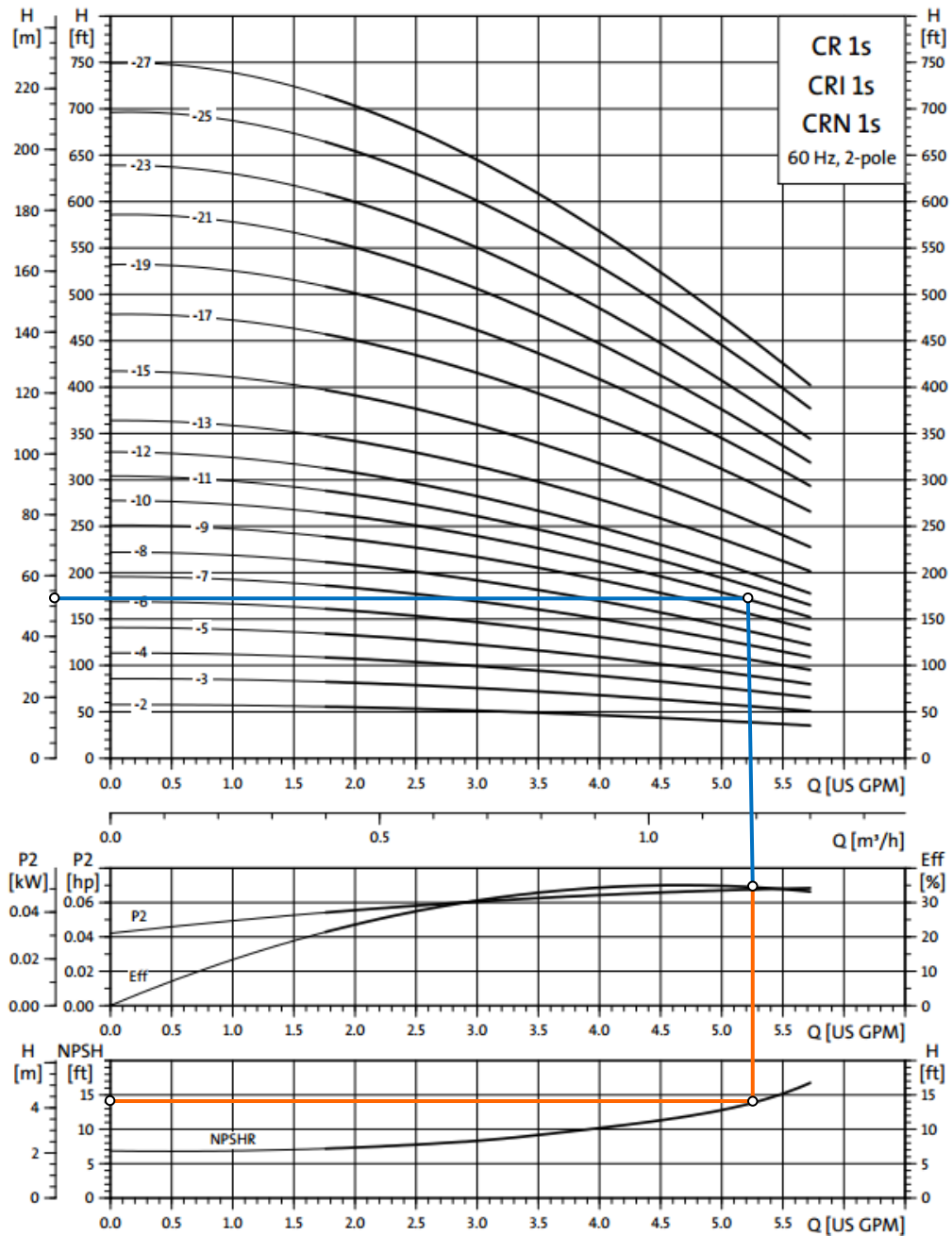
"P {bar}, T {K}, h {kJ/kg}, s {kJ/kg.K}, W{kW},"  
 "v {m<sup>3</sup>/kg}, cp {kJ/kg.K}, Q {kW}, Ti {°C},"  
 "m\_dot\_i {kg/s} and epsilon (Exergy) {kJ/kg}"

**"Comments"**

"Copy the code and paste to EES sheet."  
 "Introduce the correct units, specified above."


**A.1.3. Pump curves**

The graph shows the operation curve of the pump. In the top graph the operation point is shown, inside the graph. And. in the inferior graphs, we can appreciate the efficiency and the required NPSH.



**Figure A.1.** Pump operation curves.

**A.1.4. Pumps data sheets****Table A.13.** Pump datasheet.

<b>GENERAL</b>	Manufacturer: Number: of pumps: Service: Type:	Grundfos 3 Impulsion to small tank CR1s	
<b>OPERATION CONDITIONS</b>	Product Maximum pressure Temperature range Pressure Temperature Liquid Density Liquid Viscosity Vapour pressure Nominal flow Flow range Specific heat	Water 21 -20 to 120 1 20 998 $1.02 \cdot 10^{-3}$ 17.5 0.8 0.3 to 1.1 4.184	bar °C bar °C kg/m <sup>3</sup> Pa·s mmHg m <sup>3</sup> /h m <sup>3</sup> /h kJ/kg
<b>ASPIRATION CONDITIONS</b>	Aspiration height Aspiration pressure NPSH <sub>a</sub> NPSH <sub>r</sub> TOTAL Aspiration pressure	0 1000 7.94 4.27	m kg/cm <sup>2</sup> m m kg/cm <sup>2</sup>
<b>IMPULSION CONDITIONS</b>	Impulsion height Impulsion pressure h <sub>L</sub> elbow 90° h <sub>L</sub> check valve h <sub>L</sub> ball valve h <sub>L</sub> inlet h <sub>L</sub> outlet h <sub>L</sub> friction h <sub>L</sub> TOTAL TOTAL Impulsion pressure	0.5 6000 0.04 0.17 0.19 0.03 0.05 0.23 0.70	m kg/cm <sup>2</sup> Units m 2.00 m 1.00 m 1.00 m 1.00 m 1.00 m m kg/cm <sup>2</sup>
<b>REQUIREMENTS</b>	Pump type Maximum efficiency Power range Frequency	Centrifugal 35 0.37 to 1.1 60	% kW Hz
<b>MATERIALS</b>	Pump cover Driving Ace Flange	Stainless steel AISI 304 Steel GG25 Steel F124 Oval	
	<b>UNIVERSITAT ROVIRA I VIRGILI</b> <b>Escola Tècnica Superior d'Enginyeria Química</b> <b>Departament d'Enginyeria Química</b>		Comments:





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