Universidade do Minho
Escola de Engenharia

Pedro Miguel Gonçalves Ferreira

A study of mechanical properties of multilayer polymer sheets containing a recycled layer

December 2015

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A study of mechanical properties of multilayer polymer sheets containing a recycled layer

Master Thesis
Integrated Master on Polymer Engineering

Work settled under supervision of:
Prof. Doctor Eric Lafranche
Prof. Doctor Carla Isabel Martins

December 2015

DECLARAÇÃO

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Número do cartão do cidadão: 13735583

Título da dissertação: **A study of mechanical properties of multilayer polymer sheets containing layer**

Designação do mestrado ou do ramo do Conhecimento do doutoramento:
Ciclo de estudos conducente ao Grau de Mestre em Engenharia de Polímeros

Orientadores: Eric Lafranche e Carla Isabel Martins

Ano de conclusão: 2015

Universidade do Minho, ___/___/______

Assinatura: ____

ACKNOWLEDGMENTS

I would like to thank first and foremost to my supervisors, Doctor Eric Lafranche and Doctor Carla Isabel Martins, for their confidence, support and validation throughout the work.

Moreover, I thank the two schools: University of Minho in Portugal (international relationship services), Professor Fernando Moura Duarte and École des Mines de Douai in France which enabled it possible for me to attend in this internship. In addition, financing offered by the École des Mines de Douai and Erasmus + program which contained a preponderant paper to render it possible might be referred as well to be grateful.

Finally, I would like to thank Carlo Anzgoti, Pierre Bernard, Jean Claude and Catherine Douchain in France and Mauricio Malheiro in Portugal, for their help while the experimental work.

RESUMO

No presente trabalho, foi proposto um estudo referente às propriedades mecânicas de uma folha plana co-extrudida, contendo três camadas, na qual uma destas inclui material reciclado. A implementação de materiais reciclados na co-extrusão de filme tubular ou folha plana tem aumentado em resposta ao aumento de resíduos de plástico armazenado nos centros de reciclagem, os quais ficam acumulados. Além disso, resíduos plásticos à espera de um tratamento pode ser considerado um problema para o ambiente. Sendo isso uma grande preocupação para ser resolvida nos próximos anos. No entanto, esse tipo de plástico que tinham sido em épocas anteriores derretido para produzir folhas e filmes podem apresentar diferentes propriedades mecânicas após consequente processo de extrusão a devido à reciclagem.

Durante este trabalho de pesquisa, propõe-se misturar polímeros reciclados com polímeros virgens para compreender a influência do material virgem na melhoria das propriedades mecânicas. Como esses polímeros contem o grau de viscosidade original, então ele pode ser considerado como uma possível solução para resolver este tipo de problemas. Para provar a sua viabilidade, bem como a tentativa de melhorar os resultados, os ensaios mecânicos foram realizados comparando estudos teóricos e práticos.

As preocupações relacionadas com a utilização de 100% em polímero reciclado na camada do meio durante o processo de extrusão de filme foi observado e foi abortada como sendo uma missão impossível. O filme possuía espessura irregular e buracos ao longo da sua largura. Assim, o segundo método adotado foi o de prosseguir com o estudo para a co-extrusão de uma folha plana, incluindo o polímero reciclado na camada central, misturado com o polímero virgem para facilitar o processo. Além disso, a fim de observar o efeito do polímero virgem, as amostras foram caracterizadas de acordo com as suas propriedades mecânicas (ensaios de tração e ao rasgamento).

Como conclusão, os resultados obtidos podem ser considerados importantes para os próximos estudos relacionados às técnicas de processamento, tais como, co-extrusão implementação materiais reciclados entre as camadas.

Keyword : recycled material, co-extrusion and tearing tests

ABSTRACT

In the present work, a study referring to the mechanical properties of multilayer films including a recycled layer has been proposed. Implementing recycled materials within the sheet and film co-extrusion increased as a response to the increasing in plastic waste stored in the recycling centers, which are crammed. In addition, those plastics parts expecting a treatment might be considered a problem to the environment. And it is a major concern to be resolved for the upcoming few years. Nonetheless, those sort of plastics which had been in former times melted to produce sheets and films may present different mechanical properties after another extrusion process.

During this research work, it is proposed to blend recycled polymers with virgin polymers to understand the influence of the virgin material on the improvement of the mechanical properties. As those polymers contain the original viscosity grade, then it might be considered as being a possible solution to resolve this type of problems. To prove its viability as well as attempting to improve results, mechanical experiments were conducted comparing theoretical and practical studies.

Concerns related to the using of 100% in recycled polymer in the middle layer during the film extrusion process was observed and it was aborted as being an impossible mission. Film possess irregular thickness and holes. Thus, the second method adopted was to proceed with the study of co-extrusion sheet including recycled polymer within the layers of virgin polymer can be possible to make. Moreover, in order to observe the effect of the virgin polymer, specimens were characterized according to their mechanical properties (tensile and tearing tests).

As conclusion, results obtained might be considered as important for the upcoming studies related for the processing techniques, such as, co-extrusion implementing recycled materials between layers.

Keyword : recycled material, co-extrusion and tearing tests

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LIST OF ABBREVIATIONS AND SYMBOLS

P - Pressure

σ - Stress at maximum force

d – thickness of the sheet

ΔH^0 - Enthalpy of fusion for the LDPE 100% crystalline (J/g)

ΔH^c - Enthalpy of fusion for the composite measured in the DSC (J/g)

CI - Confidence interval

DOE - Design of experiments

DSC - Differential scanning calorimetry

LDPE FF39 – Polyethylene of the grade FF39

R - Contribution by the experimental results

R' - Contribution by the theoretical values

Xc - Cristallinity degree

Variable a – Virgin LDPE debit

Variable b – Recycled LDPE content

Variable c – Temperature of the calendars rollers

Variable d – Extrusion temperature of the recycled material

Variable e – Extrusion temperature of the virgin material

Variable f – Tear ratio applied by the puller rollers

Interaction factor A – Interaction between virgin recycled LDPE debit and recycled LDPE content

Interaction factor B – Interaction between recycled content and temperature of the calendar rollers

SUPERIOR INSTITUTIONS PRESENTION

1. UNIVERSIDADE DO MINHO

The University of Minho is currently among the most prestigious institutions of higher education in Portugal, and it contains also gradually come to assert itself on the international scene.

Founded in 1973, the University has two major poles: the campus of Gualtar in Braga, and the campus of Azurém in Guimarães.

The University of Minho is structured upon a matrix-based organisational model, which promotes interaction among its units, with a view to completing the projects that embody its mission and objectives while ensuring efficient use of its resources.

The University of Minho offers a wide range of courses corresponding to the 1st, 2nd and 3rd cycles of studies leading to undergraduate (BA/BSc), graduate (MA/MSc) or doctorate (PhD) degrees.

In addition to these cycles of studies, the University also administers a variety of courses that do not aim at above mentioned degrees, including specialised training courses (corresponding to the 2nd cycle) and advanced study courses (corresponding to the 3rd cycle).

2. ÉCOLE DES MINES DE DOUAI

École des Mines de Douai is a member of the GEM network (Groupe des Écoles des Mines). A federation of 7 institutions (Albi, Alès, Douai, Nancy, Nantes, Paris, Saint Etienne) enabling a stronger international approach and a better efficiency on the international scene. The GEM network works on 5 big issues: international matters, education and e-learning, research, mutual education offers and industrial matters.

The École des Mines de Douai was founded in 1878 by a presidential degree's. At that time, it trained qualified staff for mining activities. In 1965, the School turned into an Engineering College, no longer dedicated to mining but to industrial activities. In 1980, after an always faster evolution based on adaptation, innovation and anticipation, the École des Mines de Douai became the École Nationale Supérieure des Techniques Industrielles et de Mines (ENSTIM).

The École des Mines de Douai is a Public Institution depending on the French State, especially on the Ministry of Industry.

Higher School of Engineering and a Research Centre from the French Ministry of Industry, École des Mines de Douai focuses on:

- Higher Education:
- The education of multidisciplinary engineers;
- Adult vocational training for the needs of Industry and Administration;
- High level specialization (engineers, specialized masters, professional masters, research masters, PhD studies);
- Research and technology transfer;
- Company creation;

1 INTRODUCTION

In the present work, a study referring to the mechanical properties of multilayer films/sheets including a recycled layer has been proposed. As in recent years, the multilayer polymers films using more than two layers have been implemented on a large scale to promote new properties to the plastics parts including recycled materials among these layers to solve materials requirement and environment impact, companies and governments have been together to solve it. However, it requires funds and a previously study which aims to discover the behaviour of the recycled material tending into account a few processing conditions which may influence stability on the film bubble and crystalline microstructure developed at the interface of the layers. These conditions contain a preponderant role to the mechanical properties which have been proposed to be evaluated using specific norms.

As packaging market contains economically a preponderant role in some countries and packaging production level implementing polymers materials is growing up on a large scale, it allows increasing the requirement in materials. Therefore, recycling materials has been studied to prevent environment impact and demand in materials to the packaging companies. Recycling materials include a few disadvantages, such as loss properties and instability for reprocessing time. Nevertheless, advantages may be further than disadvantages. Otherwise these materials have not been applied to make it.

A DOE (Taguchi design of experiments) was previously performed and a bibliography research was previously composed with the ultimate goal of establish factors and factor levels which may contain further relevance to the mechanical properties that were proposed to measure. Mechanical experiments which have been proposed are tearing, tensile and study of the strength of the sample to prevent tearing propagation. Besides, in addition rheological measurements were performed which allow discovering properties of the recycled material, which contains a preponderant role while material is being transported through the extrusion machine to the die. In the walls of die exists shear stress which is related to the viscosity of the polymer and it may influence mechanical properties. The changing of molecular orientation and the residual stress may create weaknesses to the mechanical response on service. In addition, it may influence homogeneity and properties between surfaces of different layers which could be expected to promote poor adhesion properties. These topics will be developed carefully in the state of art chapter.

Recycled materials were obtained using packaging of care products, such as, for example shampoo bottles. Rheological measurements were performed since properties of those materials were uncharted. The presence of soap may be considered as containing one important role to the mechanical and rheological properties creating heterogeneities in the matrix of the new granulated and fragmented polymer. Furthermore, bottle and cap own different polymers which had been reprocessed and blended, being these materials PP and LDPE or HDPE. Aspects as this one may be considered as holding one influence to the mechanical results of the film, since, these are incompatible polymers. Granules of recycled material owns an acceptable rheological behaviour and may be used to extruder, however recycled material in fragments may not be utilized to extrude, since, viscosity described as a function of the shear ratio is definitively non linear.

As it has been referred, a DOE was previously designed and used to test tubular extrusion process. Then, beyond these processing tests it was realized that these materials could not be extruded using ideal conditions. The extrusion bubble could not sustain its form during making process. As result, it did not allow the production of tubular films possessing the required properties. Figure 1 represents a picture of the bubble being extruded. In this case, film had been extruded using virgin LDPE. Therefore, the bubble had remained stable until the recycled material had started being extruded. Nevertheless, it does not allow to understand the instability on the bubble of recycled materials. Bubble deforms easily and the size is not constant. As result, the production of tubular film was aborted.

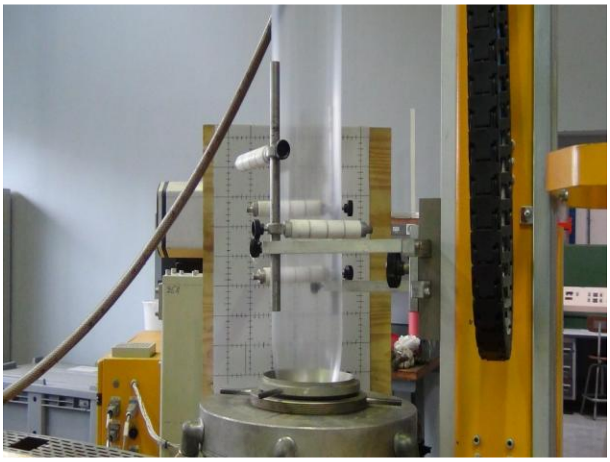


Figure 1- Bubble extruded scheme of virgin LDPE

The figure 2 represents a sample of multilayer tubular film extruded using a recycled layer (recycled material in granules). As result it allows to understand miserable properties of the film which still contains solid material in the matrix that was not melted. As conclusion, it was suggested that another material should be present on the recycled granules and consequently was proved by the DSC experiments. In addition, obtained form of the bubble was not proper, tending into account the dimensions required which were not reached. Furthermore, holes over the thickness were also detected.

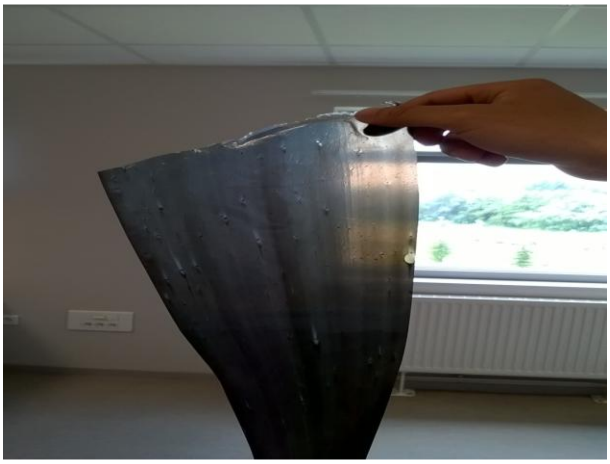


Figure 2 - Multilayer extruded film tested containing recycled layer

Thus, as tubular film extrusion had not been possible to produce, another processing technique was suggested. And such process proposed allows using the same extrusion machines and same processing transformation. However, a new die, cooling and tear system should be implemented. The new technique applied to succeed the tubular film extruding a layer containing recycled material was the planar sheet extrusion process. In this case a sheet form is obtained as shown on Figure 3 and better properties are obtained, becoming possible to make the mechanical characterization of the samples using recycled material in granules in the central layer.

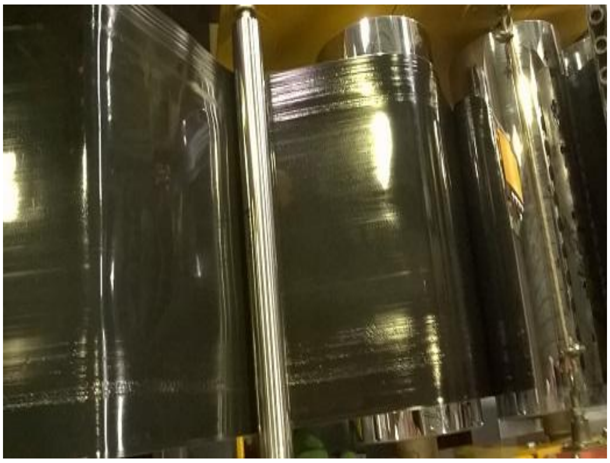


Figure 3 – Planar extrusion process

2 STATE OF ART

1. ECONOMIC AND ENVIRONMENT CONTEXT

Multilayer extrusion process or co-extrusion using recycled materials to produce tubular film or planar sheet for the packaging market has been proposed to reduce environment impact of household waste as ecological notion. High consumption of plastics inevitably leads to the production of large amounts of plastic waste especially because a substantial part of produced plastics is used for packaging parts. Recycled thermoplastics used for the packaging market have been increasing and LDPE, PP, HDPE and PET are frequently the materials recycled and used to producing packaging parts in higher proportions [1].

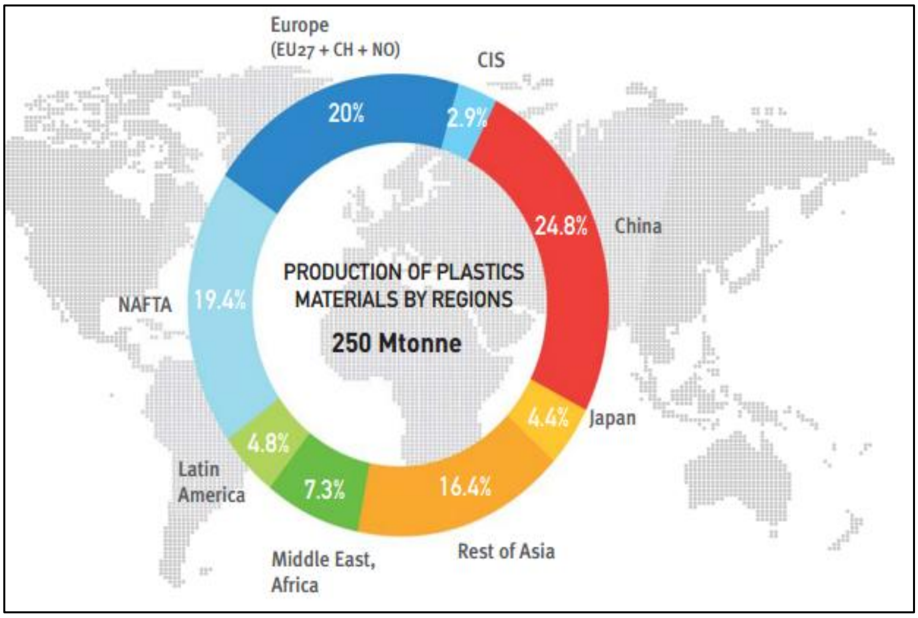


Figure 4 - Plastics materials production around the world 2013 [1]

Polyolefins (LDPE, HDPE, PP) are the main type of thermoplastic used throughout the world in such applications as bags, toys, containers, pipes (LDPE), housewares, industrial wrappings and film, gas pipes (HDPE), film, battery cases, automotive parts and electrical components (PP). Lonely in Western Europe approximately 21.37 million tons of these three polymers have been consumed each year, representing an amount of 56% of the total thermoplastics expended [2]. In addition with China and Nafta countries, these three regions spend more than 60% of the plastics consumption around the world (Figure 4). Addition polymers (like polyethylene) in contrast to condensation polymers (poly (ethylene terephthalate) (PET)) cannot be easily recycled by simple chemical methods [2].

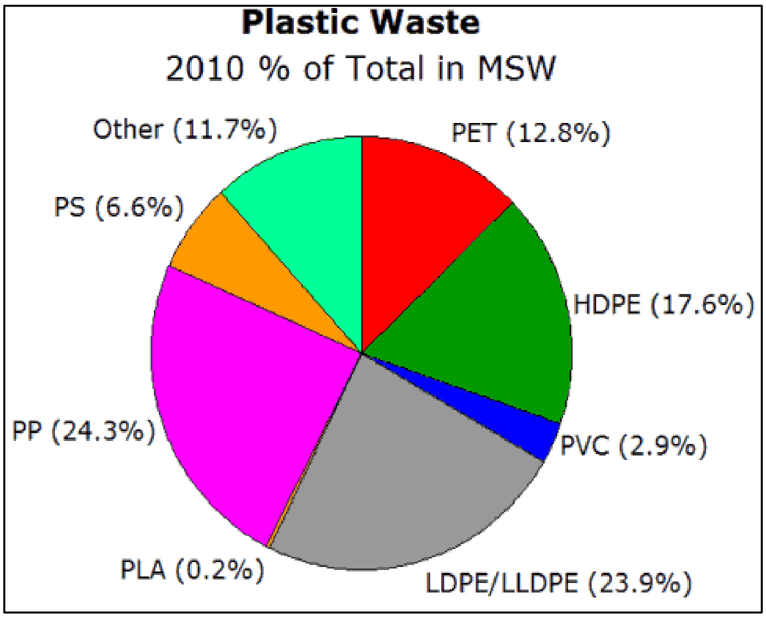


Figure 5 - Plastics waste produced in municipal solid waste (MSW) [1]

As a conclusion, the ultimate aim is not the requirement of saving natural resources as crude oil, because only 4-8% of crude are used for polymer production. But then, increases the content of plastics waste recovered to make new parts and as result to reduce the quantity of polymers waste impact. The Figure 5 represents the quantity of municipal waste produced on 2010 by some plastic materials. Per year, recycled materials is gaining ground on the markets as shown on the figure 6. Permeability, mechanical and in some situations thermal properties are the major points in this type of packaging when tubular film and planar sheet are implemented [3].

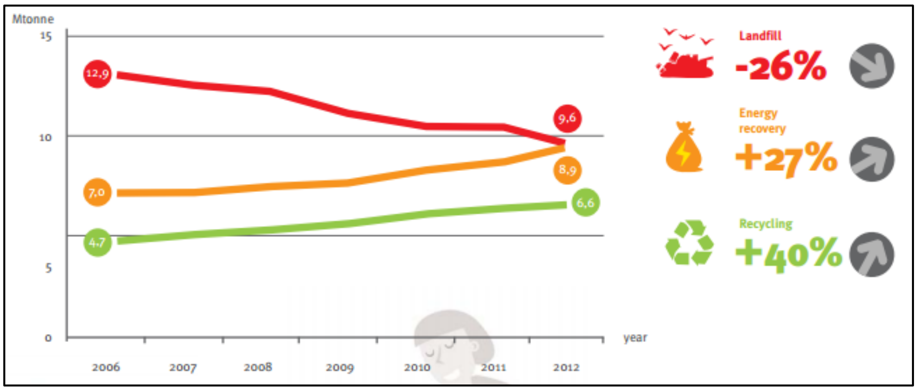


Figure 6 – Total plastics waste recycling and recovery 2006-2012 [1]

Economically, it allows decreasing the costs to produce parts. Nonetheless poor mechanical properties obtained (in some cases) is sustaining this idea working slower and forcing researchers to spend more time to discover new methods. Separation and reprocessing of the single polymeric material is also an important and interesting step [3].

In Europe, the using of planar sheet co-extruded to thermoformed packages is highly advanced, and multilayer sheet containing as many as five or six layers have been produced for applications requiring good thermoform ability, heat seal ability, stress crack resistance, barrier to gases and water vapor, and low taste and odor transfer. They have been used to package margarine, cakes, pickles, mayonnaise salads and other similar products [4]. As well to outdoor application are now being implemented such window shutters, house trim and recreation vehicles.

In Europe, Germany appears as the country where sheet extruded is more used for the packaging market (Figure 7). Yet in Germany is the Country where the polymers recycling is regulated by several legal standards [3]

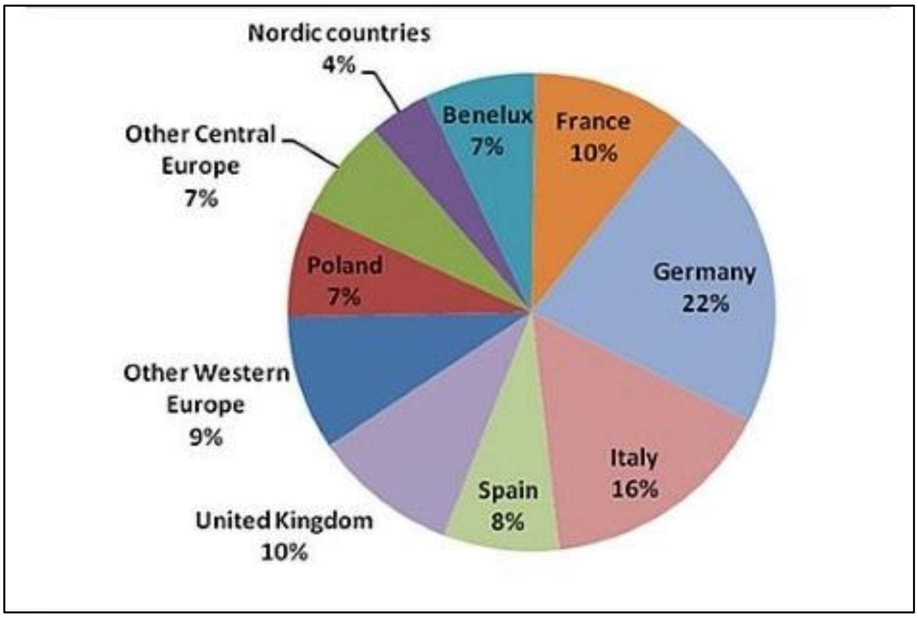


Figure 7 – Usage statistics of extruded sheets in Europe [4]

European commission proposed annex in some directives on July 2014. It concerns recycling and other waste-related targets in the EU Waste Framework Directive 2008/98/EC, the Landfill Directive 1999//31/EC and the Packaging and Packaging Waste Directive 94/62/EC. It aims render Europe into a circular economy, boost recycling, secure access to raw materials and create jobs and economic growth [5]. Below, aims of this directive are described:

- Recycling and preparing for re-use of municipal waste to be increased to 70% by 2030.
- Recycling and preparing for re-use of packaging waste to be increased to 80% by 2030, with material-specific targets set to gradually increase between 2020 and 2030 (to reach 90 % for paper by 2025 and 60% for plastics, 80% for wood, 90% of ferrous metal, aluminium and glass by the end of 2030);
- Phasing out landfilling by 2025 for recyclable (including plastics, paper, metals, glass and bio-waste) waste in non hazardous waste landfills – corresponding to a maximum landfilling rate of 25%;
- Measures aimed at reducing food waste generation by 30 % by 2025;
- Introducing an early warning system to anticipate and avoid possible compliance difficulties in Member States;
- Promoting the dissemination of best practices in all Member States, such as better use of economic instruments (e.g. landfill/incineration taxes, pay-as-you-throw schemes, incentives for municipalities) and improved separate collection;
- Improving traceability of hazardous waste;
- Increasing the cost-effectiveness of Extended Producer Responsibility schemes by defining minimum conditions for their operation;
- Simplifying reporting obligations and alleviating burdens faced by SMEs;
- Improving the reliability of key statistics through harmonised and streamlined calculation of targets;
- Improving the overall coherence of waste legislation by aligning definitions and removing obsolete legal requirements.

2. RECYCLING PROCESS MODELS TO REPRODUCE LOW VISCOSITY POLYETHYLENE

There are some models which are used to recycle materials recovered from the waste such chemical recycling, mechanical recycling and down-cycling. Mechanical recycling or physical recycling occurs when plastic is ground down and thus reprocessed and compounded to produce a new component that may or may not be the same as its original use. In the chemical recycling the polymer is turned back into its oil/hydrocarbon component in the cases of polyolefin's and monomers in the case of polyesters and polyamides, which can be used as raw materials for new polymer production and petrochemical industry, or into the pure polymers using suitable chemical solvents [6].

Chemical recycling includes pyrolysis, glycolysis, alcoholysis and hydrolysis. Generally, cleavage of the polymer chains results in chemical products of lower molecular weight distribution or even in the repetitive unit (monomers) for another complete cycle of polymerization. In the literature results have been mentioned on the glycolysis of poly(ethylene terephthalate), alcoholysis of polyurethanes, hydrolysis of nylon 6, and pyrolysis of polypropylene [7,8-9]. Nevertheless, both incineration and chemical recycling have been distinguished by the fact that the value added during polymerization is missed. At current polymer prices this comprises a major disadvantage [10]. Down-cycling consists in a molten mixture of plastics extrusion into a final form. Articles obtained by this method can be bulky. However they demonstrate mediocre mechanical properties. This fact strongly limits their applicability. Moreover, for successful application in practice, then choosing compatibilisers should not be expensive and recycling technology should not be sophisticated.

In some investigations [11], recycling models of waste products based on LDPE, HDPE and PP have been observed using two different methods: the traditional method of dissolution/reprecipitation and the more challenging technique of pyrolysis. The first belongs to the mechanical recycling techniques while the second to the chemical/feedstock recycling. For the first technique, polymer can be separated and recycled using a solvent/non-solvent system. In view of this proposition, different solvents/non-solvents had been considered and examined at different weight percent amounts and temperatures using either model polymers as raw material or commercial waste products (packaging film, bags, pipes, and food-retail products). This technique has been widely used by Papaspyrides et al. [12–14] and other researchers as well [15]. Furthermore, catalytic pyrolysis was carried out in a laboratory fixed bed reactor using as raw materials either model LDPE, HDPE and PP or waste products based on these polymers. Using thermal treatment under usually inert conditions a high-quality product may be obtained from a vast number of different polymers [3]. All compounds in the gaseous and oil fractions from pyrolysis had been identified. Conclusions have been greatly encouraging concerning alternative techniques of waste polymer recycling. Instead, thermochemical recycling techniques like pyrolysis are being proposed as process producing a series of refined petrochemical products and particularly of a liquid fraction similar with that of commercial gasoline [16].

Melt reprocessing of low density polyethylene various phenomena, such as shear modification (disentanglement), chain scission, gel breaking and chain recombination by free-radical oxidation and crosslinking, have been referred [9, 17, 18, 6]. Quite a few advantages related with this technique are the low cost of operation and easy process because it's a mechanical process that normally consists of contamination removal by sorting and washing, drying and processing. In contrast, as usually occurs, a quite few disadvantages exist and one of them is the contaminants in the matrix which are related with the function of the packaging. These contaminants promote scission of the molecular chains. Then washing is an important step since it will decrease the effect of these contaminants and those steps will not interfere with the molecular weight of the recycled polymer [19].

3. MULTILAYER SHEET EXTRUSION PROCESS

Multilayer extrusion sheets includes merely one stage on whom two or more melted polymers, using different extrusion machines, are extruded into one single matrix to form multilayers structures such films and sheets [20]. These technique allow avoiding some complex process such lamination and covering. Materials holding different properties are combined in different or equal contents. Thickness may be used to control mechanical properties, since materials with greater influence therein may be extruded in highest contents and layers can be used to give colors as well [20].

A typical extrusion line used to produce samples of sheet contains two or more single or double screw extrusion machines, one flat die which converts each circular flow coming from each extrusion machine in one rectangular flow owning the sheet form, chill rollers and a puller-rolls system. This die possess one advantage related to the coextruded capacity of polymers from many widely different rheological properties. However its limited to three layers and much longer than that operating difficulties might appear [4]. Puller rollers possess one prominent aspect as improving mechanical properties since it allows increasing molecular orientation in the flow direction when rollers speed is modified to high values. By the other side, chill rollers have also an important paper in the mechanical properties, it allows increasing or decreasing degree of crystallization depending on the temperature involved and polymers matrix (semi-crystalline or amorphous). Sheets contain better optical properties than film extruded once chill rollers have been implemented, then cooling process is promoted by contact and its further regular than using air. Likewise, the temperature of the place contains an important role as well, in consideration that it will cool the film or sheet by convection or radiation.

There are two types of sheets, low thickness and high thickness, depending on the application. The difference between film and sheets is not formally established. Though sheets until 200µm may be assumed as film and much thicker than that are considered sheets. Nevertheless, it's difficult to distingue since the process is similar. Sheets getting high thickness may be used for the thermoforming. As one example to make yogurt pots, and low thickness are used to make coverings for greenhouses.

Multilayer sheet and film extrusion process appear as techniques used to combine recycled and virgin materials. Even in low quantities, depending where the materials are employed, in tons of production it can be converted on interesting numbers as a reduction of the waste impact to the environment.

4. PERMEABILITY OF MULTILAYER FILMS

The permeation of the gases and water depends on uncertain factors such thickness and permeability of the polymers utilized in films or sheets. Permeability can be described as a function of crystallinity tending into consideration that those are related with each other (Equation 1) [8].

$$Flux = P(p1 - p2)/l \tag{1}$$

Where P is permeability, p1 and p2 is the partial pressure of the diffused gas on two sides of the film and l is the thickness.

Permeability can be also described as a function of solubility coefficient and diffusion coefficient (Equation 2) [8].

$$P = D * S \tag{2}$$

In the multilayer extrusion each polymer display its own values of D, S and P. The permeability is calculated tending into account various series of resistance. In the steady state, the diffusion flux is uniform though the film. However the concentration on diffused gas exhibits a discontinuity at each interface.

Table 1 - Approximate oxygen transmission rates through several plastics [8]

Resin	Permeability at 23° C
HDPE	150
LDPE	420
PS	260
PP	150
Rigid PVC	14
Polyester	7
Nylon	2.6

Degree of crystallinity and morphology of crystallization may be also considered as a function of permeability in polymers. Samples containing less thickness may containing much instable values of permeability [8].

5. Effects of the processing variables in the extruded sheet and film

Moura Giraldi *et al* [16] studied the influence of certain processing variables as screw speed and torque on the mechanical properties of recycled PET composites. It has been detected that screw torque on its high level was the significant factor to increase the impact strength of these composites samples. By other side, screw speed was the significant factor on its high level (200 rpm) to increase the Young's modulus. Factors interaction between screw speed and screw torque was significant for the Young's modulus, decreasing it, but no interaction factor has been detected for the Impact strength. This divergent behaviour of the processing variables, screw speed and torque, on the mechanical properties have still being analyzed, considering shearing effects of the twin screw extruder in the mixing zone (kneading discs).

Thereafter, Young modulus and the Tensile strength obtained by Giraldi [16] showed an increase as a function of glass fibers content increase in screw speed.

M.Kostadinova Loulcheva [7] studied the properties of recycled HDPE. At least one extrusion had been tested. Mechanical and rheological properties had been measured and had been obtained that mechanical properties did not achieve much changing their values as a function of the number of extrusion and mainly its strongly depending on the reproducing conditions.

In the literature [4] has been referred that several flow instabilities may occur in a multilayer melt while co-extruding a plastic film or sheet. Therefore poor melt temperature and flow surging in the extruder output rates have been the reasons that cause non uniform layer thickness. Flow surging is defined as the oscillatory change in the output rate of the extruder while maintaining constant set points conditions. In addition, also differences in viscosity have been reported in those studies as having an influence in the output rates of the layers.

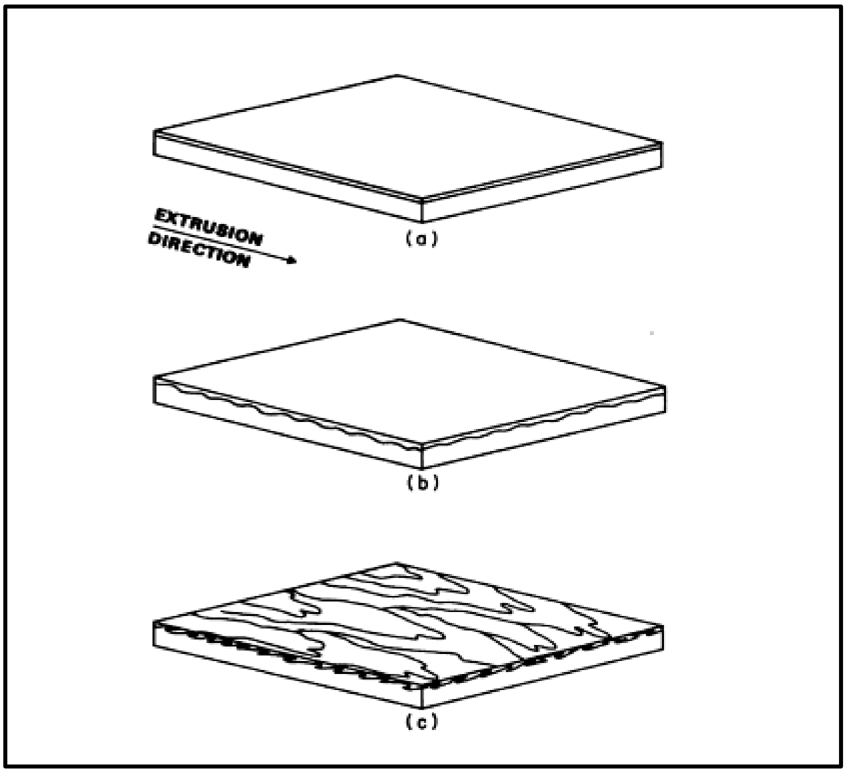


Figure 8- Illustration of film or sheet appearance under a) stable flow conditions b) incipient interfacial flow instability c) severe instability [4]

Figure 8 has been used to show how the temperature used in the die and extrusion machine might be enough to change the flow from stable to instable [4]. Critical interfacial shear stress at which interfacial flow instability may occur for a pair of polymers.

Temperature contains an influence in the behaviour of the polymer in the extrusion head. Consequently depending on the extrusion temperature and screw speed there will be a highest shear stress at the wall of the die, as a consequence of using wrongly these parameters, besides just as well, melting fracture may occurs easily in the case of the Low Density Polyethylene. The roughness of the extruded has been associated itself with a stick-slip instability of the polymer velocity near the rigid die wall [4]. Melt fracture allows creating residual stress.

Residual stress has been described as an intermolecular stress created as a result of the incorrect using of the parameters while the extrusion process. Residual stress can occur when melt temperature, cooling temperatures, rollers speed/temperatures and debit are incorrectly applied in the extrusion process. Or by other words, it appears as a consequence of the phase transition of material from melt to solid state takes place in unfavourable conditions. Specially, residual stress appears in crystalline polymers, related to the specific volume changes. In the case of semi-crystalline polymers, specific volume may change up to about 25%. Residual stress cans readily occur when semi-crystalline polymers are cooled rapidly. Then, as molecules have no time to relax, residual stress may be created between polymers molecules on the matrix. It allows decreasing mechanical properties, promoting in some instances service fatigue [21]. Residual stress may be threaded, such reheating polymer to decompress molecules. As another example, polymers extruded using melting temperatures under the allowable melting temperature will allows promoting high shear rates at die wall and barrel that will allows increasing residual stress since material has difficulty to relax his molecules and chains.

Polymers hold two components, viscous and elastic. A limit separates these two components and when polymers are cooled briefly, in some situations elastic limit cannot be reached, as result polymers molecules remain in stress state. As described in polymers literature, elastic component allows recuperating deformation suffered until this limit (normally is assumed as 2%). Exceeding elastic limit, probabilities to create residual stress have been lower. Furthermore, the movement of polymer molecules during their melting process are restricted by intermolecular interaction such conformation Trans, Gauche+ and Gauche-.

The thickness of a extruded tubular film has not clearly affected the CO2 permeability (PCO2), but the O2 permeability (PO2) tended to increase, as the thickness of the film had increased. Although, only the two largest thicknesses significantly affected the PO2 [22].

6. EFFECTS OF THE RECYCLING IN THE PROPERTIES OF THE LDPE AND OTHER THERMOPLASTICS

Reprocessing of produced waste is also known as in-house or primary recycling and represents a type of mechanical recycling where plastics are grinded and then processed through a physical process [22]. In these cases material may be exposed to excessive number of reprocessing that may affect its processability and quality of the end products.

Properties of the recycled polymers are affected by degradation from heat, mechanical stresses and oxidation during their reprocessing. Molecular structure and mechanical properties are affected. Besides, rheological properties can be also affected, what will directly affect the processibility and it requires to be considered during the following processes [22].

F. Awaja and D. Pavel [19] detected in extruded PET samples of oriented blow molding extrusion bottles that molecular orientation and crystallization are related to each other in which crystallization is promoted by orientation. The degree of orientation and crystallization affects individually and interactively a bottle mechanical, optical and barrier properties. In the study of Awaja had been detected one decreasing in mechanical properties as a function of the number of extrusions [19].

S. Chytiri and al. [23] observed that the results of tensile strength, percent elongation at break and Young's modulus did not suffered statistically significant differences between no irradiated samples containing virgin LDPE and those containing recycled LDPE as a middle implemented layer. The same was observed in irradiated multilayer structures (all absorbed doses) containing or not recycled LDPE. Chytiri had used five layers on the multilayer extrusion process as it is possible to observe (Figure 7).

S. Berlin J-J Robin and al. [24] discovered that LDPE/PP recycled blends detain mechanical properties after extrusion process using twin-screw, this technique allows improving blend between these two polymers. Nevertheless, toughness decrease and it promotes a decreasing in impact strength.

Pavel Oblak [25] studied the effect of mechanical recycling on processability and mechanical properties of HDPE. Results on rheological measurements, MFI, complex viscosity, storage and loss modulus, showed unfavourable effect of mechanical recycling on HDPE processability. He had structured the study for 30th cycles what the effects have been more observed as preponderant in the last ten cycles.

W. Nunes dos Santos and al [26] studied the effect of the recycling in the thermal properties of some polymers. Then he encountered that thermal properties in the processing region possess thermal diffusion practically identical. However, near the glass transition temperature, the variation is higher and generally recycled polymer presents a lower thermal diffusion. Reprocessing causes a strong reduction in the mean molar masses, generating significant structural differences in the recycled material in comparison with virgin PP. Yet, in the case of the Nylon 6.6, a semi crystalline engineering thermoplastic which contains lower average molecular weight, which should facilitate the crystallization process of the recycled polymer, yielding higher thermal diffusivity values.

7. CRYSTALLINITY AND MOLECULAR ORIENTATION EFFECT IN SHEET AND FILM PROPERTIES

Crystallinity owns an important role in the mechanical and barrier properties of the films and sheets. The higher degree of crystallinity promotes lower permeability (Robertson, 1993) since the crystalline regions are relatively impermeable compared with the amorphous areas. For a given penetrant molecule the rate of diffusion is governed by the nature of the polymeric chains and therefore changes in the molecular structure may influence its diffusional behaviour [23]. Chill rollers holds an influence in the degree of crystallinity depending on their temperature. As a result, crystallinity improves young modulus under glass transition temperature.

In addition, molecular orientation contains also an influence in the mechanical properties such young modulus. Young modulus is higher in the direction where molecules are preferentially oriented. In this technique, molecules suffer molecular orientation preferentially in the transversal direction promoted by puller rollers located in the end of the line. When samples are requested in this direction (transversal direction), tensile strength should be higher than in the longitudinal direction [22].

8. MOLECULAR WEIGHT EFFECT

Quite a few suggestions exist in the literature that molecular and structural factors may influence the character of the deformation process in the LDPE. For example, Steidl and Pelzbauer [27] pointed out that the molecular weight of the sample alters the shape of the force-elongation curve.

Molecular weight loss is also a problem which has to be taken into account when polymers materials are recycled. As it has been mentioned recently in the last sub chapters, molecular scission can occur, which allows decreasing molecular weight.

C. Sadrmohaghegh and G. Scott [26] studied the thermal recycling and it formed that cross-linking contains an influence in those properties. Cross-linking allows increasing molecular weight which will decrease MFI (Melt flow index). As a result, mechanical properties might be improved.

9. PRESENCE OF THE PP IN THE BLEND

The majority of the packaging products have been made using PP and LDPE, then recycling these materials may be interesting. Nevertheless, solve difficulties to separate them is also required. Furthermore, techniques that allows separating molecules becomes its expensive as it was explained in the paragraphs.

S. Berlin, J-J Robin et al [24] studied LDPE-PP recycled blends. They detected that until 30% in content of PP in a matrix containing recycled LDPE, may improve mechanical properties such young modulus and tensile strength at yield. Nonetheless, a decreasing in elongation at break and impact strength had been detected. Blend becomes much rigid, yet more fragile when breaking.

LDPE and PP are incompatible and it has been proved in many studies [8, 24, 28, 29], using microscopic and calorimetric analyses. PP and LDPE are incompatible in the melted phase and blend appears as a two phase mixture. Usually, mechanical properties are dim and it may be explained tending into account the poor interfacial bond between both phases [28]. Mechanical performances may be improved using additives as fillers or compatibilizers. Yet there is not a universal compatibilizer for all kinds of mixture.

10. CONCLUSIONS AND WORK PROGRAM

After the bibliographic research, work program and study aims are ready to be designed. The aim of the study is evaluate the mechanical and barrier properties of multilayer's polymers films of recycled and virgin LDPE. If possible, the improvement will be also taken into account.

As work program, the following steps are proposed:

- Study of the flow behaviour (Capillary Rheometry);
- Measure the melting points and enthalpy energy of the recycled materials by DSC;
- Design a plan of experiments (DOE) for sheet extrusion: a DOE containing three levels is required to make sheet samples that will be useful to study the influence of the extrusion parameters on the sheet properties;
- Multilayer sheets extrusion of materials levels;

- Mechanical tests are required to characterize there samples(Tensile tests, tearing tests and study of the tearing propagation);

- Some microscopic experiments will be proceeded to observe the thickness of the layer containing recycled material.

- Conclusions and future perspectives of the using recycled LDPE in multilayer sheets.

3 MATERIALS AND METHODS

1. MATERIALS AND EXTRUSION MACHINE USED

Materials proposed to implement in the multilayer sheet extrusion process were virgin low density polyethylene (LDPE) with reference Riblene FF39, recycled PE in granules and recycled PE in fragments. Properties of these materials are described on the Table 2. Recycled materials derive of hygienic or care products packaging

Table 2 - Materials properties

Material	Grade	Melting point (ºC)	Density (g/cm^3)	Morphology
Virgin LDPE	FF39FF	114	0,924	Semi-crystalline
Granulated PE	PEOK	132/164	not measured	Semi-crystalline
Recycled PE in fragments	PRPE	136	not measured	Semi-crystalline

Figure 9 and Figure 10 represent the recycled PE in granules and fragments respectively. Recycled PE was reprocessed to obtain the granules (Figure 9) or crushed using a mechanical method to obtain the fragments (Figure 10). In this recycled material can exist low density polyethylene, high density polyethylene and polypropylene because the kind of referenced packaging is produced using these polymer materials. Some tests (DSC and Capillary Rheometry for example) may be proposed to confirm this argument.

Figure 9 – Granules of recycled PE

Figure 10 – Fragments of recycled PE

3 layer sheets were proposed to be produce by tubular/planar extrusion process, with outer layers containing virgin material and middle layers containing virgin and recycled material in different contents. Three blends using recycled material and virgin material have been proposed according to the table 3. The production using 100% in recycled material was tested, however it was not possible to make because of the recycled material properties which contains more than one polymer, changing the rheological properties. These properties hamper the contact between the layers in the exit of the main die.

Table 3 – Blends proposed to the central layer

Blend (Numbering)	Recycled Content in the LDPE matrix (%)
1	25
2	50
3	75

As a first aspect, the blends which were proposed to utilize on the sheet extrusion do not require any special treatment. Therefore, blends may be ordinarily prepared using contents as described on the Table 3. In addition, the recycled and virgin material may be immediately introduced into the extrusion machine which is responsible to melt, to blend and to move by the screw until the die these materials.

Figure 11 – Extrusion machines (25A, 25B and 30A)

The figure 11 shows the extrusion process line which contains three extrusion machines 25A (extrusion 1), 25B (extrusion 3), 30A (extrusion 3) and a die which is responsible to converge circular flow in rectangular flow and generate a sheet form. (Dimensions of the die: 1mm of slack and 35cm of with). This line was designed and made by a German brand (DR COLLIN GMBH).

In addition, a cooling system has to be present in this line which includes a preponderant role on the optic and structural properties. This system possess a calenders system which has a prominent role in the mechanical properties of the sheets tending into account the influence to modify macrostructure which is influenced by the time and cooling temperature. In this system exist puller rolls which are responsible for varying molecular orientation which contains a preponderant role to the mechanical properties of the sheets. Lastly, at the end of the line, there is a stocking roll which is responsible to roll up the sheet. Puller and stocker systems are represent in the Figure 12.

Figure 12 – Tearing and cooling system

A panel control is available on the machine which allows controlling some parameters, such as, extrusion pressure, temperature of the barrel, rotation screw speed, temperature of the extrusion die and temperature of the main die which contains rectangular form to produce this sheet. Each extrusion machine owns this control panel (Figure 13).

Figure 13 – Control panel

2. Rheological characterization

Rheological experiments tending the capillary rheometry as method to measure the rheological properties of the materials were proceeded. These experiments were made to measure the rheological constants (n and k) and obtain the flow curves (Shear rate as a function of viscosity). Where n and k are the rheological constants of each material and this function represents viscosity as a function of the shear rate. where K is the consistency index (Pa.s) and n is the power-law index. The importance to calculate these constants and obtain the flow curves is related with the preparation of the extrusion process and the understanding of the planar sheet properties obtained. In other words, evaluate the shear rate as a function of viscosity may be considered as an important aspect to be taken into consideration during the extrusion process. If the materials suffer a significant difference in shear rate during the exit of the die, mechanical properties obtained can be effected negatively. As the technique used in this case is the multilayer tubular/planar extrusion, rheological properties are quite important to evaluate the stability of the process. The using of extrusion temperatures which are not appropriate can originate some rheological defects, such as, shark skin and residual tension. Following, the proceeding of the Bagley correction used to determinate the rheological properties of the materials will be explained.

1.From the geometry present on the extrusion machines dies and screws, the model used to represent dies and screw flow is even the same as the one approached in the capillary Rheometry. Where apparent shear rate near the wall is defined by equation 3.

$$\gamma_{w\,(app)} = \frac{4 * Q}{\pi * R^3} \tag{3}$$

2. Afterwards and through Rabinowitsch correction to apparent shear rate near from the wall is defined by the equation 4, applied to non-Newtonians polymers,

$$\gamma_{w(real)} = \frac{4 * Q}{\pi * R^3} * \frac{3 + n}{4} \tag{4}$$

Q – volume flow,

R – Radius of the capillary channel,

n – Slop obtained through the bi-logarithmic relation between $\log w_{(app)}$ and $\log \tau w$.

3. Tending into account the elimination of the viscous and elastic effect in the entry of the capillary, the equation 5 is applied to and through the Bagley correction apparent shear stress is measured, $\boldsymbol{\tau}$w.

$$\tau_{w(app)} = \frac{\Delta P}{2 * L} * R$$

(5)

4. Obtaining the follow, equation 6, that express the real shear stress:

$$\tau_{w(real)} = \frac{\Delta P}{2 * \left(\frac{L}{R} + e\right)} * R = \frac{\Delta P - P_0}{2 * L} * R$$

(6)

ΔP – Pressure drop in the capillary

L and R – Capillary radius and length

e – Correction Bagley factor

P0 – Represent pressure drop in a capillary containing zero length for a given shear.

The Bagley correction for ΔPe will be explained consecutively. Unless a very long capillary is used (L/D> 100), entrance pressure drop may considerably effect the accuracy of the measurements.

The Bagley correction is used to correct for this, by assuming that this extra entrance pressure drop can be represented by an equivalent length of die, e: Three or four capillaries are used and results are plotted as DP vs L/R.

The true shear stress is:

$$\tau w = \Delta P/(2 * \left(\frac{L}{R} + e\right))$$

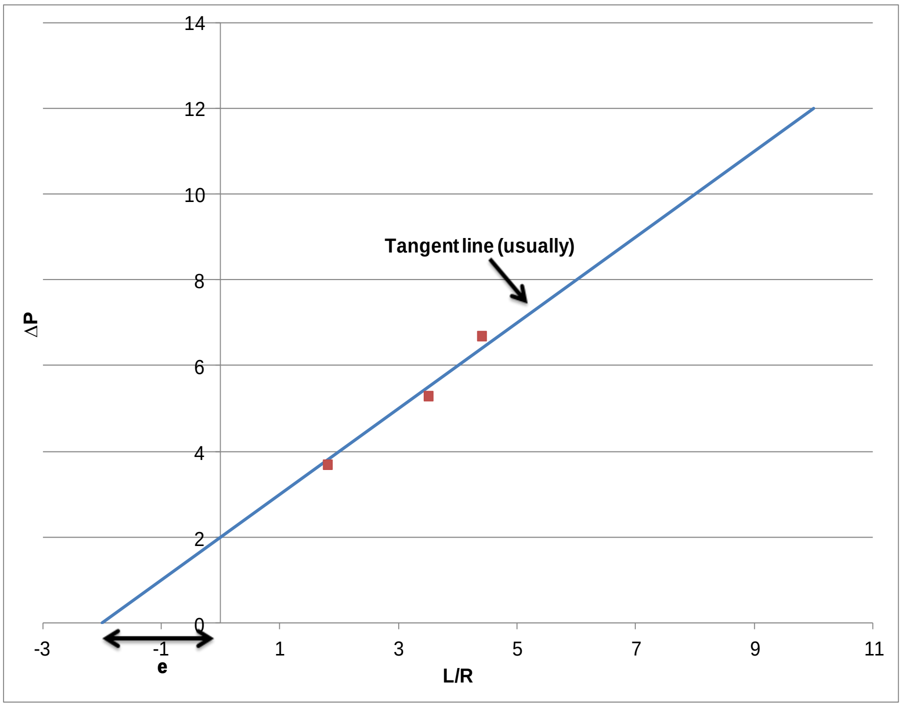


Figure 14 – Bagley correction for ΔPe

Bagley correction factor can be calculated plotting the pressure drop as a function of the L/R by each shear rate. A tangent line is used to express a linear regression and by plotting the equation y=mx+b, the value when y=0, is the value that may be taken as the Bagley correction factor.

As the last step, using all corrections made, real viscosity can be measured (Equation 7), just dividing the real shear stress calculated using Bagley correction and real shear rated determinate using Rabinowitsch correction (Figure 14).

$$\eta = \frac{\tau}{\gamma} \tag{7}$$

Unless a very long capillary is used (L/D>100), entrance pressure drop may considerably affect the accuracy of the measurements. The Bagley correction is used to correct for this, by assuming that it can be represented a extra entrance pressure drop by an equivalent length of die, e. Three or four capillaries are used and results are plotted as DP/L/R.

This viscosity value allows to describe viscosity as a function of the shear rate using different temperatures. This information is important to take to the extrusion process as temperatures and debits values. Per each extrusion machine used is required to obtain a good sheet extruded.

Rheological behaviour was measured using a rheometer of the German brand GOTTFERT and model RHEOGRAPH 2001, which is illustrated on Figure 15. It contains three heat zones, each one has been heated until the same temperature per each test. The behaviour of each polymer has been studied in three different dies. It allows knowing the polymers behaviour in high shear rate values, equals to the registered in the extrusion process.

Figure 15 – Rheometer used to the Capillary rheometry

More input information is indicated on table 4 (the die dimensions) and the input parameters on the Rheometer are also indicated on table 5. Besides input mechanical parameters, input shear rates applied by each polymer are accessible on table 6. One information which is possible to extract from the table is that two different shear rates input were introduced on the machine. The rheological behaviour of the LDPE in fragments was really instable and incontrollable. Rheological study was repeated often and changing shear rates values to reach the values which allows measuring the required values to the calculus.

Table 4 – Dimensions of the dies used

Die	Diameter (mm)	Length (mm)	Angle (º)
Low	1	10	1
Medium	1	20	1
High	1	30	1

Table 5 – Rheometer parameters input

Piston diameter (mm)	Recording interval (s)	Sensor of pressure (bar)	Pressure tolerance (%)
12	8	1000	2

Table 6 – Shear rates input

Polymer	Shear rates (s^{-1})							
Riblene FF39 and recycled granulated	3000	1500	800	500	250	100	50	20
LDPE in bits	1500	800	500	250	100	50	20	10

$$\eta = k * \gamma^{n-1} \tag{8}$$

Equation 8 represents a potential function which describes the rheological behaviour of the polymers.

The virgin material, recycled material in fragments and granules were submitted to a rheological study tending into account the following temperatures of reference, T=170°C, T=1900°C, T=210°C, T=230°C.

3. DSC (Differential scanning calorimetry) to identify the materials

Differential scanning calorimetry (DSC) has been widely used in the study of blend compatibility as well as in the determination of crystallinity in crystalline amorphous polymers blends. DSC is a relatively new technique; its name has existed since 1963, when Perkin - Elmer marketed their DSC-1, the first DSC. In all the points, it allows measuring melting temperature, glass transition temperature, crystalline phase transition temperature and specific heat or heat capacity [32].

A DSC machine is constituted by two single ovens in which the temperature gradient between the block and the oven must be high enough to obtain a precise temperature control of the sample. In each oven circulates a purge gas, usually nitrogen, with high thermal conductivity and purity with constant flow. This gas will perform an important role in monitoring the temperature of the sample. Each oven has separate temperature sensors, so that the calorific flows are provided independently to each oven. Each oven have one capsule (normally in aluminium), in one oven will have an empty capsule with a reference and in the other will have one capsule with the sample that will be tested.

To ensure the credibility of the experimental results, it is necessary to calibrate the DSC machine. In the calibration, a baseline is created which defines a heating rate without putting any material in the capsules, resulting in a variety of information from the curve, as the contamination of the device, deterioration of the thermocouples and magnitude of the background noise.

DSC is a technique in which the heat flow rate difference into a substance and a reference is measured as a function of temperature, and consequently plotted, while the sample is subjected to a controlled temperature program.

In DSC, the enthalpy change is calculated from the temperature difference between the sample and the reference. In endothermic processes (processes with energy absorption, such as melting or evaporation), the enthalpy of the system increases, while in exothermic processes (condensation, crystallization) the enthalpy (and the internal energy) of the system decreases. Similar to the SI unit for heat, the SI unit for enthalpy is J (joule) [32]

In this case, DSC analysis was performed to measure the melting point and fusion enthalpy. The melting point allows perceiving the materials which are present in the recycled material. It can be important to the extrusion process control, as one example the extrusion temperature of the recycled material.

DSC analysis was carried one time in each sample in 2 different granules, in which these granules contained different colors. The different color might mean in practice that the granule was taken from a different packaging, increasing the possibility to discover another polymer. The cuts were made in the microtome, and proximately 5mg of mass was used to the measuring. The tests were made in a PERKIN-ELEMER DSC-7, with the nitrogen as purge gas, from 25°C to 200°C at 10°C/ min.

4. MEASURE OF THE EXTRUSION MACHINE'S DEBIT

Parameters related to the extrusion process will be referenced on the chapter where the plan of experiments (DOE) might be aborted. Furthermore, in each extrusion machine different parameters may be applied since each extrusion machine contains different barrel and screw dimensions. Given that, debit of each extrusion machine have been previously measured to obtain a massic debit curve, plotted in terms of debit (Kg/h) as a function of speed screw (rpm). Moreover, different materials contains different properties and their properties were unknown. Given that, some proceedings had been adopted, being one of them to introduce material in each extrusion machine that had been previously chosen to extruded one of the materials and using the help of a chronometer, three samples were taken to measure their mass per each 3minutes of extrusion. Results of debit have been measured using three different rotation speed: those perceived between the highest speed level which can be reached by the machine and two other intermediate values. The importance to the study of the debit control is to promote a good adhesion between the layers at the end of the main die and obtain the same thickness per each layer using the same debit in each extrusion machine.

Extrusion machines geometry is different therefore to extrude the same debit in each extrusion machine, different pressures and rotation speed should be applied. In the extrusion 30A to reach to the same debit as 25A and 25B, rotation speed of the screw is relatively less.

5. DETERMINATION OF THE SHEAR RATE IN THE DIES

The shear rate contains an important role to the study of the interfacial instability between layers extruded simultaneously on the same die . The technique used to extrude the sheet possess a rectangular die that is responsible to join the three layers from each extrusion machine flow. In this extrusion process three levels of debit are used. Despite of that just one debit is required to be study and used to understand a few interfacial instabilities and residual stress that may be present on the film originating poor mechanical properties.

There are some properties which might be noted from our extrusion process, such as, pressure, geometric dimensions and debit of the conjoined. Debit was previously calculated using the same materials, curves of viscosity as a function of the shear rate were previously plotted from the rheological experiments and pressure was given by the pressure detector. Then, taking into account pressure read by the detector, debit from each debit curve, viscosity may be discovered and consequently observed on the graph viscosity as a function of the shear rate to detected respective shear rate, according to the equation 10.

$$Q\ die = \left(\frac{(\pi * R^4)}{(8 * L)}\right) * (\frac{\Delta P}{\eta})$$

(9)

Where,

Q die is the debit of the die

R is the die radius

L is the length of the die

ΔP is the pressure on the die

η is the viscosity of the material at this temperature

Difference in shear rate in the wall of the die may be used to comprehend fragilities on the mechanical tests which can be related to interfacial instabilities. As it has been previously observer these materials own a different rheological behaviour which is important to prevent encapsulation between two polymers. However, some materials can contain another behaviour which is normally applied as roughly miscible, since they have the same origin. Nonetheless a quite few content in recycled polypropylene may be observed and present on the blend which can change completely extrusion behaviour, originating encapsulation of the LDPE and consequently problems to the mechanical answer during their service.

In addition, shear rate in the die of the conjoined may be calculated using models which contains capacity to discover this value. Tending into account of geometric dimensions and debit calculated previously, then reach to the shear rate value is more simple. It will be calculated using Equation 11 and 12 and table 7:

$$\ddot{Y}a = \frac{6 * Q}{W * H^2} * \left(1 + \left(\frac{H}{W}\right)\right) * f * (\frac{H}{W})$$

(10)

$$\gamma = \ddot{Y}a * \left(\frac{2}{3}\right) * \left(\left(\frac{b'}{f'}\right) + \left(\frac{a'}{f' * n}\right)\right)$$

(11)

Table 7 – Geometric constants for rectangular dies

H/W	a*	b*	f'
0	0.5	1	1
0.05	0.4535	0.9513	0.9365
0.1	0.4132	0.9098	0.882
0.15	0.3781	0.8745	0.8351
0.2	0.3475	0.8444	0.7946
0.25	0.3212	0.8183	0.7597
0.3	0.2991	0.7954	0.7297
0.35	0.2809	0.775	0.704
0.4	0.2659	0.7571	0.682
0.45	0.2538	0.7414	0.6634
0.5	0.2439	0.7278	0.6478
0.55	0.236	0.7163	0.6348
0.6	0.2297	0.7065	0.6242
0.65	0.2248	0.6985	0.6155
0.7	0.2208	0.6921	0.6085
0.75	0.2178	0.687	0.6032
0.8	0.2155	0.6831	0.5991
0.85	0.2139	0.6803	0.5961
0.9	0.2129	0.6785	0.5942
0.95	0.2123	0.6774	0.5931
1	0.2121	0.6771	0.5928

6. DOE APPLIED TO THE MULTILAYER EXTRUSION PROCESS

The DOE (Design of experiments) is a technique used for planning experiments, i.e., sets what data, quantity and conditions should be used in a given experience. DOE owns two main objectives: obtain better statistical accuracy in response and, do it without having significant costs. In addition, it identifies critical factors which optimize the average response of a process, minimize its variability and make them much more robust.

An experimental plan is required for a subject which is proposed containing unknown conditions, the objective of the experience has to be established, the parameters which have influence to the experience and arrange them. To start with, it is required to evaluate the resources and costs and thus select the factors to study and the answers to evaluate. The experimental plan have been constructed, based on the Taguchi method, which may reduce significantly the number of experiments, preserving an important clarification.

DOE applied, to determine the optimal level of the variables, orthogonal matrices that allow studying more variables just by making some experiments. For allocation of the variables and its interactions in the orthogonal matrices, Taguchi proposes the use of the interaction tables, denominated triangular table. For each orthogonal matrix there is a triangular table.

Depending of the number of variables that should be studied, the matrix is different. For example, an L8 matrix allows studying from three until seven variables, depending of the number of interactions between the factors that is intended to study. On the other hand, a L16 matrix allows to study from seven until fifteen variables at two levels. Finally, an L27 may be used to study up to 13 factors at three levels.

The answers intended to study in this work are already established: the mechanical behaviour of multilayer extrusion sheet. Using DOE analysis becomes much more simply to encounter the extrusion conditions which may improve the mechanical performance, without performing large quantities of experiments.

The first problem is to know how many variables to study and the reasons why. As there are a considerable number in processing variables that can influence the mechanical properties of the sheet, and as it is difficult to study all of them, it has been decided to select the conditions that apparently contains more influence. To avoid the mistake of choosing the incorrect conditions, it has been decided to select a greatest range of conditions possible. Therefore, the processing conditions that should be studied have been: debit of the virgin material (Q.virg), recycled content (Rec.cont.), stretch out speed (Tear ratio), input extrusion temperature per each material (Trec and Tvirg), temperature of the rolls (Trolls) responsible to cool extruded sheet (calendar rollers).

7. PLAN OF EXPERIMENTS

Consequently, a plan of experiments is required to apply DOE. To that end, three levels for each variable have to be established, and then the Taguchi method containing 27 levels has to be implemented. Tending into account the triangular table and the orthogonal matrix, specific for this experience in particular. Combining the concepts of the orthogonal matrix and its triangular table, a table of experiments with all experiments that are required to start it have to be created. Six variables have been decided to be studied. As result, a L27 matrix may be filled as it allows studying these six extrusion parameters containing three levels, and just as well any interactions between these variables. These tables are accessible on the appendixes (Figure 32 and 33).

Consequently, after selecting the appropriated table of experiments, it is possible to construct the plan of experiments. This plan is not further than, an orderly and logical organization of the table of experiments.

Consecutively, a method that may reduce the time of the experiments proceeding might be elaborate. Therefore, it is necessary to maintain in mind facts as: cooling time of the extrusion barrel since temperature is higher than the heating time. Thus, it is important to initiate the experiments which contain lower limit of extrusion temperature because it takes more time to cool the extrusion machine than to heat. It is just one example of the principle followed.

Combination of variables are described as A and B. These combinations were considered as having effect in the extrusion process. As in this case occurs, tear ratio and recycled temperature have not been chosen since it does not causes any changing in the final properties of the sheets.

8. MULTILAYER EXTRUSION PROCESS

As of the moment that the plan of experiments is elaborated, the conditions for initiate the multilayer extrusion process are compiled. Then, the next step is to start the multilayer extrusion processing.

The DOE allows perceiving the processing conditions which allow improving various properties. In this situation, the mechanical performance, without having to produce thousands of samples in thousands of combinations of processing conditions. Therefore, just twenty seven experiments may to be produced. Samples have been produced in each experiment, and in these samples mechanical tests (tensile, tearing tests and strength to the tearing propagation behaviour) should be performed.

In the following tables 8 and 9 is shown levels applied per each experimental variable. Orderly, Q virg.and Q rec. contain as levels (1.8, 2.8 and 3.8 Kg/h), T rolls (20°C, 30°C and 40°C), Trec (195°C, 205°C and 215°C), T virg (170°C, 185°C and 200°C) and T ratio (2, 4 and 6). The debit levels (Q virg and Q rec) were chosen taking into account some experiments proceeded previously, in which polymers could be extruded using values up to this values. The T rolls took into account the typical values applied for a polyethylene as cooling temperatures. The

Trec took into account the typical extrusion temperatures used for a polyethylene and the presence in polypropylene which increased the temperature required to melt the granules. The T virg were applied tending datasheet as support. And finally, the Tear ratio which was chosen taking into consideration the capacity of the puller rollers, in which the scale is between 1 and 7.

Table 8 – Taguchi's L27 orthogonal array

	Q vir	Rec content (%)	Qvirg-Qrec	Trolls	Trolls-Rec content	Trec	Tvirg	Tear ratio
	a	b	A	c	B	d	e	f
L1	1	1	1	1	1	1	1	1
L2	1	1	1	2	5	2	2	2
L3	1	1	1	3	9	3	3	3
L4	1	2	5	1	6	2	2	3
L5	1	2	5	2	7	3	3	1
L6	1	2	5	3	2	1	1	2
L7	1	3	9	1	8	3	3	2
L8	1	3	9	2	3	1	1	3
L9	1	3	9	3	4	2	2	1
L10	2	1	6	1	1	2	3	2
L11	2	1	6	2	5	3	1	3
L12	2	1	6	3	9	1	2	1
L13	2	2	7	1	6	3	1	1
L14	2	2	7	2	7	1	2	2
L15	2	2	7	3	2	2	3	3
L16	2	3	2	1	8	1	2	3
L17	2	3	2	2	3	2	3	1
L18	2	3	2	3	4	3	1	2
L19	3	1	8	1	1	3	2	3
L20	3	1	8	2	5	1	3	1
L21	3	1	8	3	9	2	1	2
L22	3	2	3	1	6	1	3	2
L23	3	2	3	2	7	2	1	3
L24	3	2	3	3	2	3	2	1
L25	3	3	4	1	8	2	1	1
L26	3	3	4	2	3	3	2	2

Table 9 - Plan of experiments according to Taguchi method

	Q virg	Rec content (%)	Qvirg/Rec content	T rolls	Trolls/recycled content	Trec	Tvirg	Tear ratio
	a	b	A	c	B	d	e	f
L1	1.8	25	1	20	1	195	170	2
L2	1.8	25	1	30	5	205	185	4
L3	1.8	25	1	40	9	215	200	6
L4	1.8	50	5	20	6	205	185	6
L5	1.8	50	5	30	7	215	200	2
L6	1.8	50	5	40	2	195	170	4
L7	1.8	75	9	20	8	215	200	4
L8	1.8	75	9	30	3	195	170	6
L9	1.8	75	9	40	4	205	185	2
L10	2.8	25	6	20	1	205	200	4
L11	2.8	25	6	30	5	215	170	6
L12	2.8	25	6	40	9	195	185	2
L13	2.8	50	7	20	6	215	170	2
L14	2.8	50	7	30	7	195	185	4
L15	2.8	50	7	40	2	205	200	6
L16	2.8	75	2	20	8	195	185	6
L17	2.8	75	2	30	3	205	200	2
L18	2.8	75	2	40	4	215	170	4
L19	3.8	25	8	20	1	215	185	6
L20	3.8	25	8	30	5	195	200	2
L21	3.8	25	8	40	9	205	170	4
L22	3.8	50	3	20	6	195	200	4
L23	3.8	50	3	30	7	205	170	6
L24	3.8	50	3	40	2	215	185	2
L25	3.8	75	4	20	8	205	170	2
L26	3.8	75	4	30	3	215	185	4
L27	3.8	75	4	40	4	195	200	6

9. DECISION MATRIX

The step which follows it, is the construction of the decision matrix. The first step is to follow the table of experiments, and completing the table tending into account the results which have been measured in the mechanical tests; the second step is to calculate the average of all those values that are in the positive position and also the average of all values that are in the negative position. After this, it is necessary to do the subtraction of the average between the result of values in the positive and negative position, and if this subtraction gives a positive result, the higher value is the best to improve the mechanical properties. On the other hand, if the result of the subtraction is negative, the smaller value of the variable is the best to improve the mechanical properties. This analysis is then done in a summary table, called decision matrix.

A numerical model needs to be implemented to study the influence level of each variable present in the plan of experiments. The preceding information related to the average can be used to measure the numerical coefficients of the model. The numerical model can be written in the form:

$$y = X + [ai] * Qvirg + [bj] * Rec.cont. + [ck] * Trolls + [dl] * Trec + [em] * Tvirg + [fn] * Tear\ ratio + Qvirg * [\alpha jk] * Rec.cont. + Trolls * [\beta kl] * Rec.cont. \quad (12)$$

Where:

y is the response of the plan

X is the average of all the results (mechanical in this case)

a, b, c, d, e and f are the lines of the matrix corresponding with the effect of the factors

α and β are the matrix 2x2 corresponding with the effect of the interactions

(13)

The plan of experiments selected contains the Taguchi methodology, existing a table L_{27}, in which the columns correspond with the factors and lines of experiments.

In terms of analyzing results, the level of each model coefficient allows to determine by comparison the influence of the factors in their domain of variation.

After the DOE analysis, the rather levels of all variables which leads to a better mechanical and barrier behaviour may be discovered. It represents that when performing an experience that combines all levels of improved performance of all variables, the resultant parts will have the best possible properties. This combination is called optimal set of factors.

10. MECHANICAL PROPERTIES MEASUREMENTS

In order to study the influence of each content of recycled LDPE on the sheet properties, some mechanical tests were performed. The mechanical tests were carried in a universal mechanical testing machine: Instron Model 1185 5500R. Tensile, tearing and strength to the tearing propagation behaviour tests were proposed to characterize the twenty seven samples of extruded sheets present in the plan of experiments.

In order to measure tensile strength, tests according to the French standard norm ISO 527-3:1995 has been applied. In addition, these specimens contain normalized dimensions: length 150mm, width 25mm and thickness within 0.24 and 0.9mm which are described on the figure 15. Ten essays have been attempted for each set of samples, and these are longitudinal and transversal direction. Transversal direction implies that the samples are taken tending into account an angle of 90° with the direction of the flow and longitudinal direction might be the same as the direction of the flow. These experiments was monitored at 200mm/min. In this case, maximum stress was the variable used to characterize the samples in terms of tensile strength as a function of the displacement.

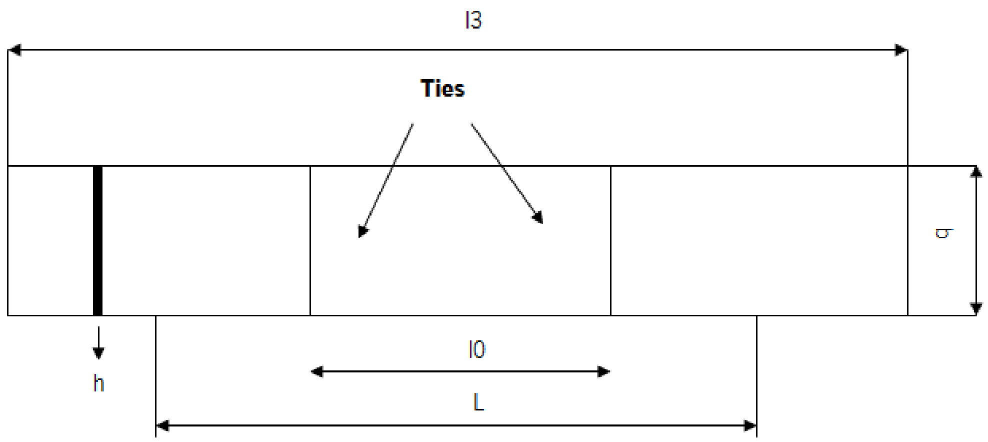


Figure 16 - Schematic representation of a tensile test sample

b – Width of 25mm;

L0 – distance between marks of 50mm;

L – Distance between arms of 100mm;

l3 – Total length of 150 mm;

h – Thickness less than 1mm, depending of each sample, there is one different thickness

Consequently, tear testing method was proposed to be proceed, in this case, also a French standard norm has been implemented, NF T 54-140 October 1984 ISO 6383/1. In this experiments, at least five samples in each direction (longitudinal and transversal) might be prepared and studied to measure the tear strength. Prevent cuts in the borders of the sample, plain surface and measure the thickness are requests of the standard norm, such a way to prevent mistakes during the experiment. Sample dimensions are shown in the figure 16. In order to obtain good results, the preparation of the samples, test speed and air conditions are properties that can change the tear strength results, since it requires attention to prepare them. Load as a function of time are the variables which might be plotted on the graph to describe results. Consequently, the tearing force is obtained tending into account the graph present by the annexes (Figure 28), where one average is calculated tending into consideration the interval where force is constant. Tearing strength is calculated devising tear force per thickness of each sample. The experiment speed was 200mm/min.

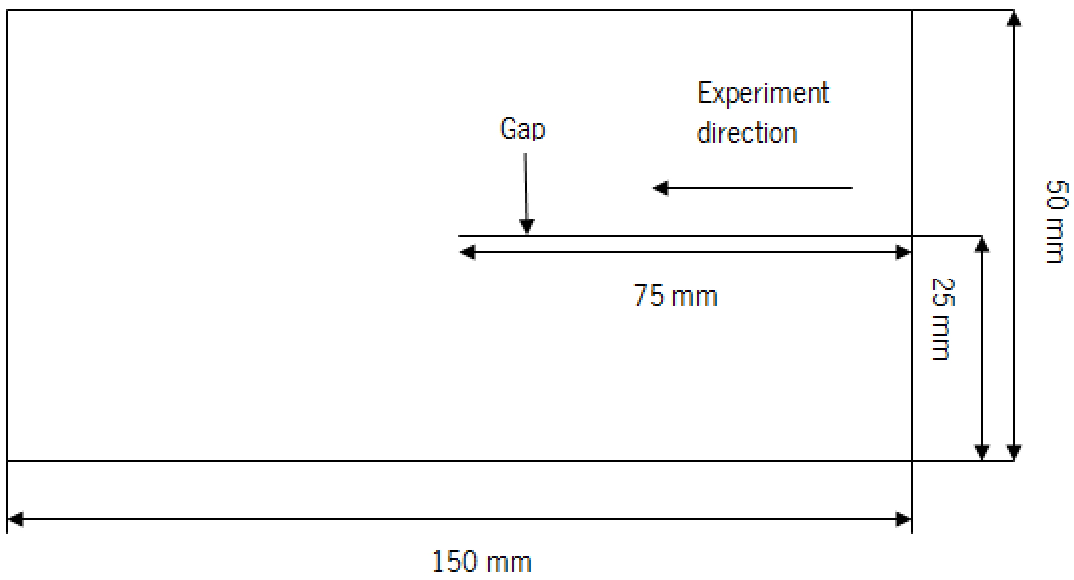


Figure 17 – Tearing test sample

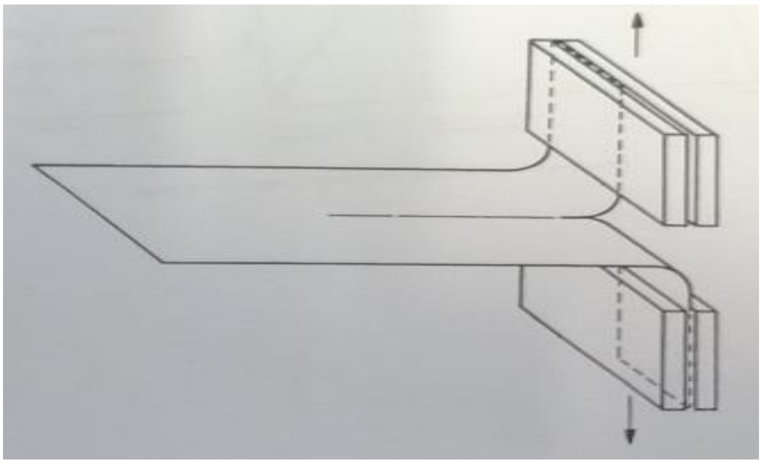


Figure 18 - Method of attachment in the arms machine

Another mechanical test has been performed, such a way to understand the behaviour of the samples when these are submitted to a tearing force. It is used to determinate the strength to the tearing propagation during the experiment, and consequently, it allows to understand the influence of the thickness. However, the difference between the thickness of each sample cannot exceed 10%. Results are plotted in force as a function of time.

It also succeed a French Norm (NF T 54 – 108). Experiment speed applied was 100 mm/min +- 10 mm/min. Sample dimensions are exposed in the figure 18.

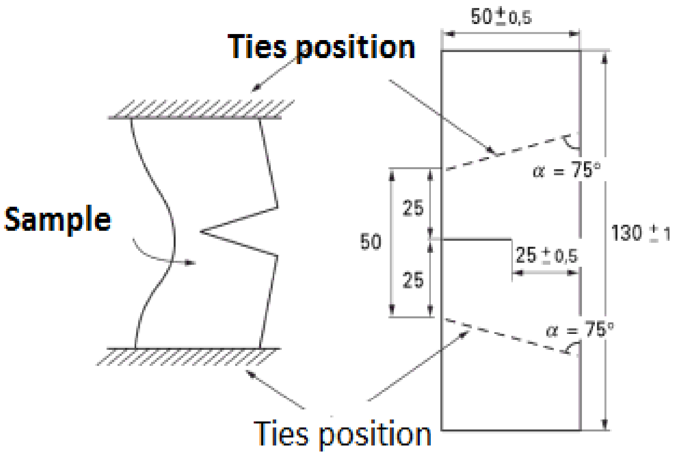


Figure 19 – Study of the Strength to the tearing propagation behaviour

As LDPE is an anisotropic material, then ten samples are required in each direction. As an important note, it might be referred that longitudinal direction is the direction of the melted flow and transversal direction is the surface which makes an angle of 90° with the direction of flow. In addition, the experiment speed used to characterize these samples was 100 mm/min.

Tensile tests, tearing tests and study of the strength to the tearing propagation behaviour tests were proposed as required to characterize these twenty seven sheet samples present in the Taguchi plan of experiments. Those mechanical tests allow understanding the influence of the extrusion parameters in the final properties of the sheets.

11. OPTICAL MICROSCOPY

Optical microscopy is often the first technique used to examine or compare products or prepare small samples before further analysis is performed. In the optical microscopy, digital images of samples are obtained, and this allows understanding the microstructure and the product property relationships.

The samples for the microscopic analysis requires a detailed preparation to be possible the observation. The preparation of these samples can be done in several ways, but usually resorts to the microtome for do micrometric cuts.

Canada balsam can be used between the glass slide and cover glass to facilitate the process and increase the lifetime of the sample.

4 RESULTS AND DISCUSSION

1. RHEOLOGICAL CHARACTERIZATION

The virgin material (Riblene FF39, LDPE) viscosity as a function of shear rate is plotted on figure 20 for the four temperatures of reference. The equation which describes the rheological behaviour of non-Newtonian-materials tending into account the potential law is described downwards as Equation 14 for this material. Tending into account this equation, n and k of the Riblene FF39 are 0.4 and 12401 (Pa*s) respectively. The rheological behaviour of the virgin LDPE is considered as typical because the curve obtained is linear (viscosity changes linearly as a function of shear rate) and the viscosity decreases as a function of temperature linearly as well.

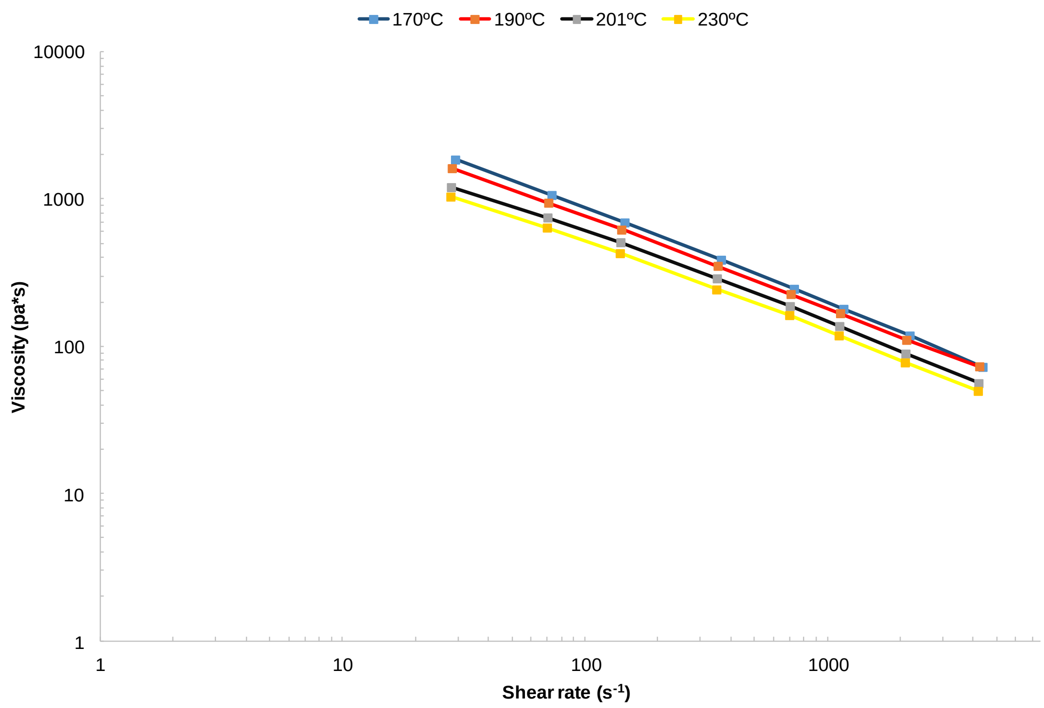


Figure 20 – Curves of Riblene FF39 material

$$y = 12401 * x^{-0,628} \tag{14}$$

Rheological results corresponded to the granules of recycled material are plotted on Figure 21.Even that another material had been supposed to be present in the blend, rheological properties obtained for this material may be acceptable to become possible the extrusion in normal conditions. In other words, it is possible to introduce into the extrusion machine disposable on the processing laboratory. The curves shown that the viscosity between 170°C and 210°C does not change as changed using virgin material. Then, it can be used to enhance the idea that another polymer is present in the blend. In addition, the polymer included with the polyethylene in the granules contains a highest melting temperature which influence the viscosity of the recycled material because it requires more temperature to melt. The rheological constants of the recycled material which can be measured are n=0.43 and k=7473 (Pa*s). As this recycled material was reprocessed, the heterogeneity between the distribution of the molecules present in the blend decreased, improving its properties.

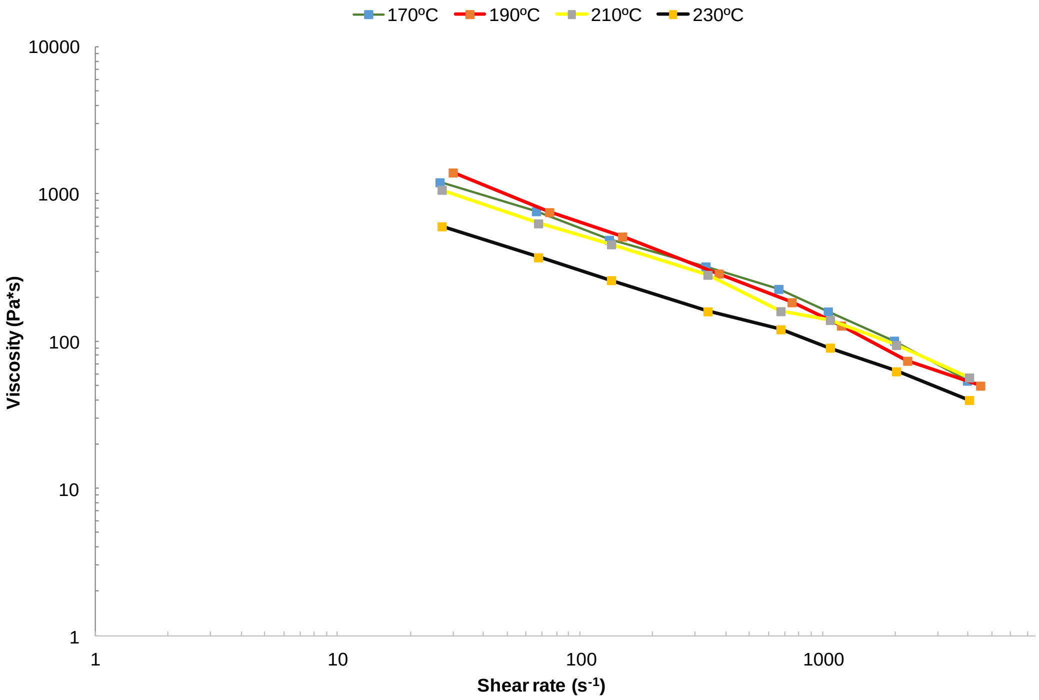


Figure 21 – Flow curves of granulated material

$$= 7473,2 * x^{-0579} \tag{15}$$

Rheological results of the recycled material in fragments allow understanding what definitively prevented to render possible extrusion process using this material. As it has been referred in the introduction, the recycled material was taken from the shampoo bottles and other packaging of care products which contain at least two polymers, one in the bottle and another one in the cap. In consideration of the results , it has been detected that the recycled polymer contains instable and non-linear rheological properties. Results were reached tempting often the same parameters and different dies to measure at least seven points. Tending into account these results, it was suggested that another polymer could be present in the fragments of the recycled material, then this material was eliminated of the study and consequently was not introduced in the extrusion machine because of the non-linear rheological behaviour. In which the case of the fragments possess greater importance in terms of results, since these fragments did not traversed a reprocessing step. As a suggestion to solve this problem, the separation methods and create a purification method should be introduced or changed in order to improve the quality of the recycled material.

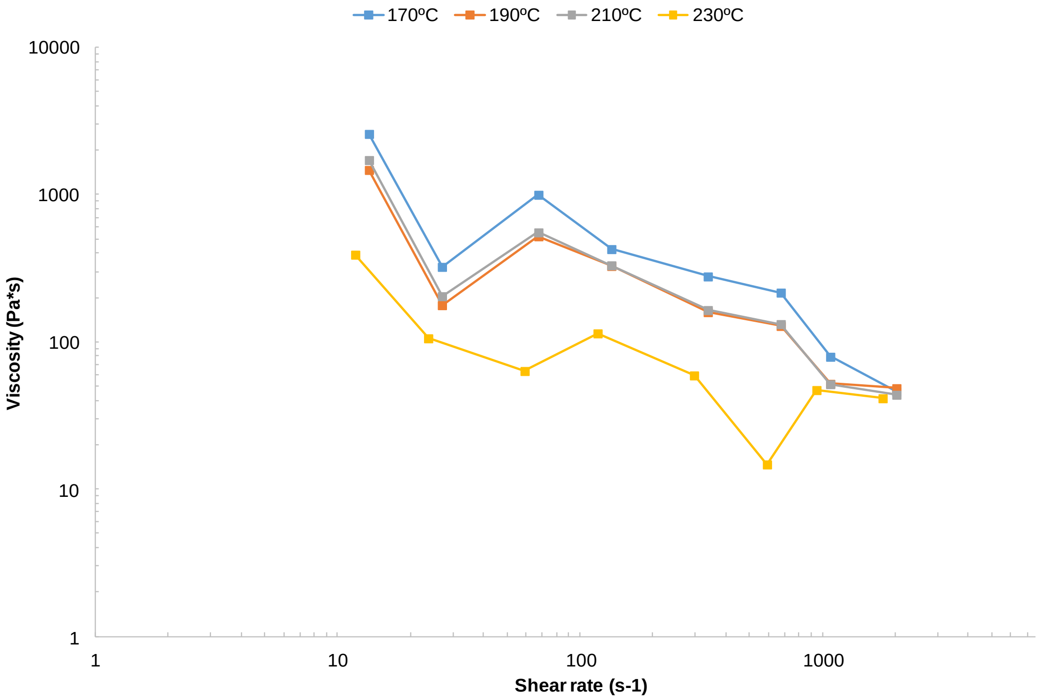


Figure 22 - Flow curves of fragmented material

$$\eta = 584 * x^{-0,413}$$

(16)

Table 10 – Rheological constants of the materials

Materials	n	K (Pa*s)
Riblene FF39	0.4	12401
Granules of recycled material	0.43	7473
Fragments of recycled material	0.59	584

2. Debit of the extrusion machines

Curves related to the extrusion debit of each machine using virgin LDPE (25A and 25B) and recycled PE (30A) are plotted on the Figure 23. Tending into account the capacity of each extrusion machine to debit material, then debits of these three extrusion machine should owns the same debit, adjusted by the rotation screw speed.

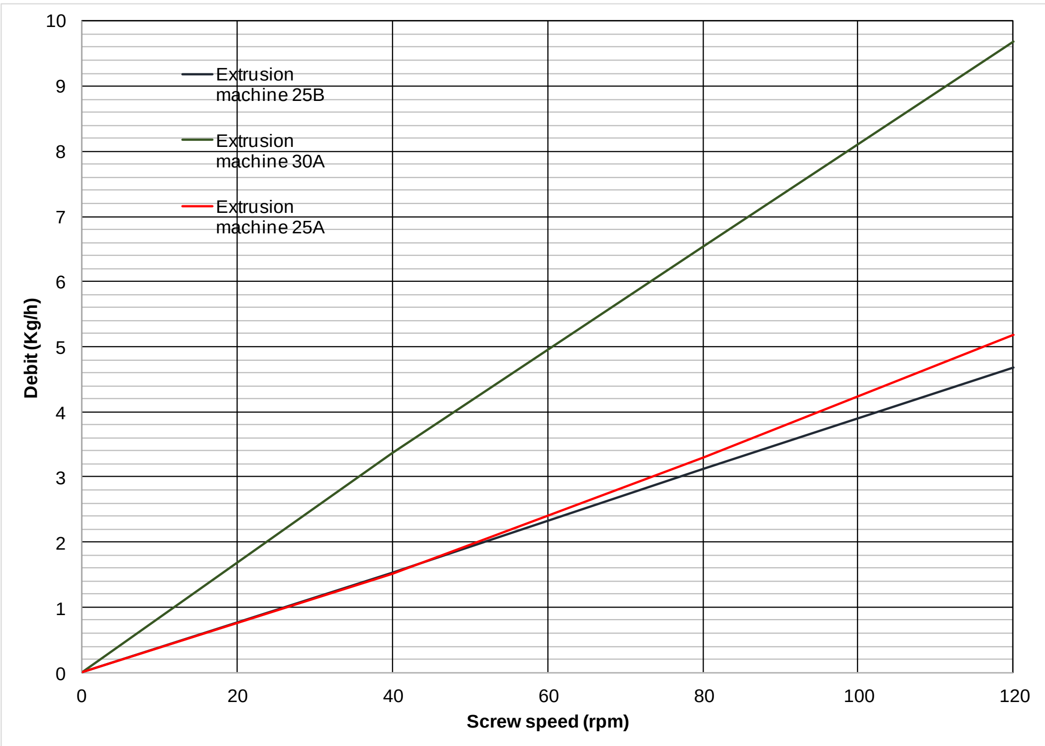


Figure 23 – Curves of debit of the extrusion machines

Results demonstrated that screw dimensions contains an important role to the capacity to debit of the machine. Extrusion machine 30A was the extrusion machine containing more capacity to debit, then the screw speed used on the machine 30A to extrude should be less than other extrusion machines. These curves were taken to encounter points of intersection between extrusion machines to use the same debit tending into consideration rotation screw speed.

3. SHEAR RATE IN THE DIES OF THE EXTRUSION MACHINES

Tending into account the equations 9, 10, 11 and processing parameters, shear rate in the die may be measured. These results contains a considerable paper to the evaluation of the rheological behaviour in the dies which may be responsible by origin interfacial instabilities and consequently poor mechanical results. On table 11 are represented results of the shear rate in the rectangular or principal die tending into account the debit applied on the extrusion machine and geometrics constants.

Table 11 – Shear rate in the profile die

Shear rate of the main die						
Débit (m^3/s)	Débit (Kg/h)	W (m)	H(m)	Υ (s-1)	Ϋ (s-1)	Visc (Pa*s)
0.00	1.80	0.35	0.01	12183.86	8563.57	37.87
0.00	2.80	0.35	0.01	18952.67	13321.11	24.35
0.00	3.80	0.35	0.01	25721.48	18078.65	17.94
0.00	1.80	0.35	0.01	12183.86	8563.57	47.18
0.00	2.80	0.35	0.01	18952.67	13321.11	30.33
0.00	3.80	0.35	0.01	25721.48	18078.65	22.35

On table 12 are available shear rates calculated in the circular dies of each extrusion machine, tending into consideration debit, geometric constants and pressure given by the pressure detector.

Table 12 – Shear rate in the circular die of the extrusion machine

Extrusion machines 25A and B shear rate in the die using virgin ldpe						
Débit (m^3/s)	Débit (Kg/h)	Pressure	L (m)	R (m)	Visc (Pa*s)	shear rate (s-1)
0.00	1.80	11800000.00	0.15	0.01	158.58	58254.09
0.00	2.80	11200000.00	0.15	0.01	96.76	35544.87
0.00	3.80	13900000.00	0.15	0.01	88.49	32504.85
Extrusion machine 30A shear rate in the die using recycled ldpe						
Débit (m^3/s)	Débit (Kg/h)	Pressure	L (m)	R (m)	Visc (Pa*s)	shear rate (s-1)
0.00	1.80	11800000.00	0.15	0.01	169.56	62289.01
0.00	2.80	11200000.00	0.15	0.01	103.46	38006.85
0.00	3.80	13900000.00	0.15	0.01	94.61	34756.27

Differences in shear rate may not be considered huge as expected as it has been previewed, nevertheless a difference exist and it can be noted to be implemented in the conclusion of the mechanical results.

4. DSC RESULTS TO IDENTIFY THE MATERIALS

Such as referred previously, DSC was used to measure melting point and fusion enthalpy of the granulated and fragmented recycled LDPE. Heating speed implemented to study these samples was 10°C/min. Samples are heated two times to take the melting point from the second heating because the first heating takes into account the influence of the making process. In the first heating, samples contain some dirtiness and crystalline zones which were formed during the making process. Then, a second heating allows measuring melting point in a proper way and physically more acceptable.

Figure 24 and 25 depicts the DSC curves of recycled LPDE. From the plot, It is suggested that another polymer is also present in fragmented or granulated recycled LDPE because two melting points are found. The second melting point is seen as being typical from the virgin Polypropylene. The presence of the PP allows increasing melting temperature of the granulated LDPE. In the followed figure 24, it is shown DSC curve of the granulated recycled LDPE. Tending into account the obtained value and some typical polymers DSC peaks, it may be considered that this material is polypropylene. The figure 25 shown the DSC curve of the recycled LDPE in fragments, in which the behaviour is similar, proving the suppositions about the material which could be present in the blend.

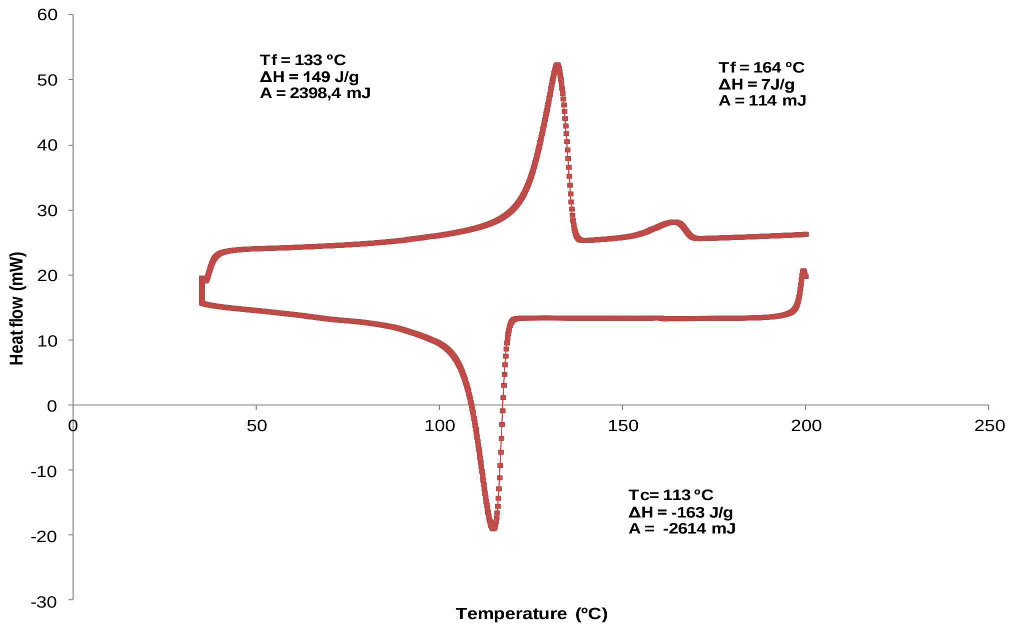


Figure 24 – DSC curve of the granulated recycled PE

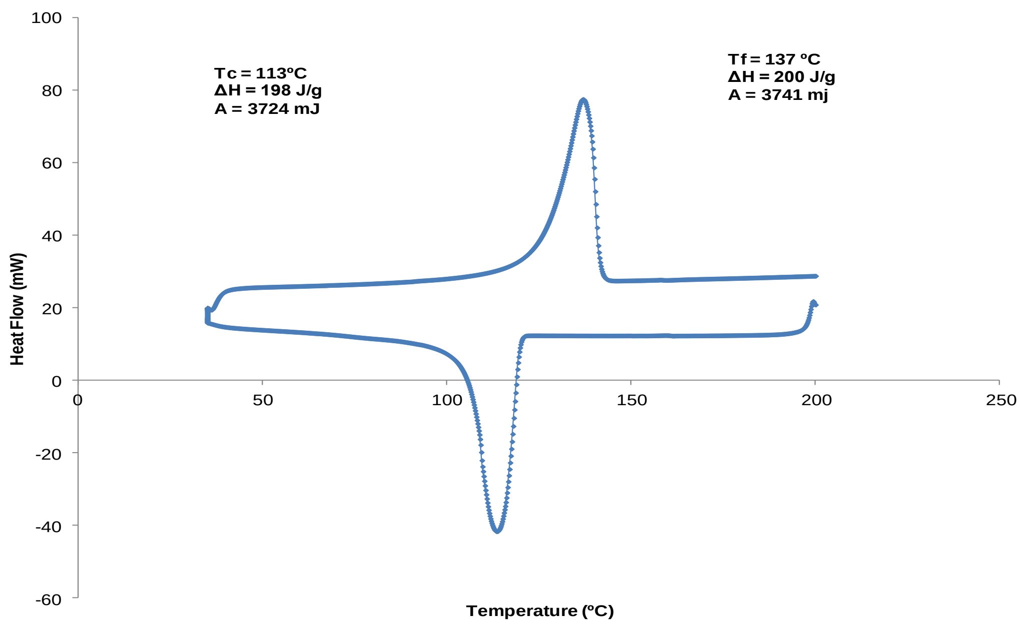


Figure 25 - DSC curve of the fragmented recycled PE

Second peak is lower than first peak, however it is considered important to reach until the material which is also present with the LDPE. Then, it indicates that content in PP is small. As result, it can cause problems to the extrusion process. Results are also tabulated in the followed tables 13 and 14.

Table 13 – Crystallization and melting temperatures

Material	Tf (ºC)	Tc (ºC)
Granulated	133	113
Fragmented	137	113

Table 14 – Enthalpy of crystallization and melting

Materials	ΔHF(j/g)	ΔH c(j/g)
Granulated	164	163
Fragmented	200	198

5. INFLUENCE OF THE PARAMETERS IN THE MECHANICAL PROPERTIES

To determine the optimal set of the processing conditions, it is required to relate the results of mechanical analysis with the processing conditions and levels. For this, it was created a decision matrix, as was explained, on the experimental procedure.

The first parameter to consider is if the difference between the higher and lower levels considered for the variables have definitively a significant influence on the modulus. Therefore, weather the difference between the maximum and minimum values recorded is higher than 10% the confidence interval (CI). It indicates that the levels selected had been sufficient to vary the property. In this case, the following equation can be used:

$$CI = (Value_{maximal} - Value_{minimal}) * 100 \quad (17)$$

Where:

- CI - Confidence interval

- $G_{maximal}$ - Maximal value for modulus
- $G_{minimal}$ - Minimal value for modulus

The study of the effect of the processing conditions for the maximum stress in the longitudinal direction have been attempted and the results are represented in the figure 26.

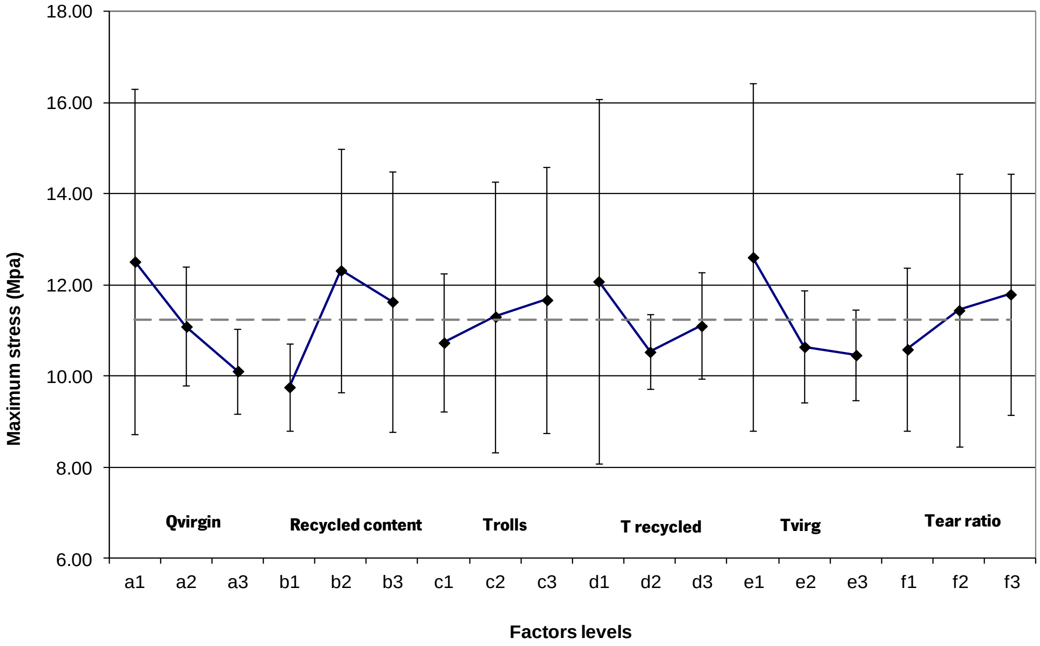


Figure 26 - Effect of variables in the maximum stress for the longitudinal direction

In this case the confidence interval is: $CI = (12{,}61 - 9{,}76) = 2{,}85\%$. The result obtained is 2,85%, which means that the results can be accepted.

Analyzing the results in the figure 26, it is observed that the variables a, d and e have a negative effect on the stress at maximum force and the variables b, c and f have a positive effect. The variables a ,d and e in lowest level, and the variables c, and f in the highest level and b in the intermediate level contains definitely more contribution to have a greater maximum stress. These results may be explained based on the theoretical concepts referred in the state of art chapter.

The debit of the virgin material (a) contains great influence on the modulus 6,16%, but it occurs in the lowest level. When the debit in virgin material is lowest, maximum stress reached are greater. As material extruded is fewer, it allows increasing molecular orientation, as result of the lower thickness which enable to stretching much more the polymer molecules. As a positive consequence, force required to break the extruded sheet increases as a function of the decreasing in virgin material. When the viscosity difference is large between the layers, viscous encapsulation was a very strong effect in both the circular and square channel. As properties are difference between recycled and virgin, as demonstrated in the rheology results, then including less virgin material , encapsulated material diminish noting that it is caused by the presence of PP in the recycled material, as result interface deformation decreases just as well residual stress and mechanical results obtained are rather improved.

Calendar rollers temperature (c) holds not a preponderant influence on the maximum stress, about 0,94%. In addition, this variable affects particularly degree of crystallization, promoting more or less nucleus since material is semi-crystalline all the time, depending merely on the cooling time and temperature. As result maximum stress in the longitudinal direction increases as a function of the increasing in calendar rollers temperature because crystalline zones formed during cooling time are definitively more. These organized zones promotes more orientation which improves tensile stress.

The tear ration, (f), owns a considerable influence on the maximum stress, containing a percentage about 1,64%, when it is in a highest level. Increasing tear ratio promotes an increase of the maximum stress. Molecular orientation is frozen by the stretching out promoted by the puller rollers, then it allows increasing the maximum stress as a function of tear ratio.

The temperature of the recycled material (d) possess an important influence on the modulus, about 2,58%, since temperature of the recycled material have a direct effect on the material structure and just as well on the interfacial properties. If the recycled temperature is high (205°C), it may allows an increasing in degree of crystalinity which promotes much more capacity to support tensile stress. However, presence of those two materials containing rheological properties out-of-balance, nothing may be preview to conclude. Results shows a initial decreasing from the lowest temperature to the medium temperature which may be related to the interfacial instability promoted by the increasing in viscosity difference between these materials. Though, from medium temperature to highest temperature, a short increasement have been observed which can be related to the decrease in recycled viscosity to virgin viscosity which cans decreases interfacial instability and, consequently, critical shear stress between layers what will improve tensile properties.

The temperature of the virgin material (e) contains a negative effect which may be explained by the viscosity of each material. As a large difference in rheological materials properties exists, an increase in extrusion temperature allows extending this difference. Then interfacial instability related to the critical shear stress increases. which promotes a decrement in the tensile properties. Virgin temperature owns considerable influence to the tensile stress, about 5,98%.

The effect of the other variables on the maximum tensile stress is about 25% and for this the influence of these can be considered important. Yet, residual influence is too high.

Thereafter, interactions between parameters have to be observed and discussed. At first, interaction between virgin debit (a) and recycled content (b) have been proposed to be discussed (figure 27). In this case, any interaction is observed which indicates that recycled content and virgin debit interaction does not influence tensile stress at maximum force in the longitudinal direction. As it have been observed on the figure 27, those two parameters contain a different influence then therefore cannot interact in one attached way.

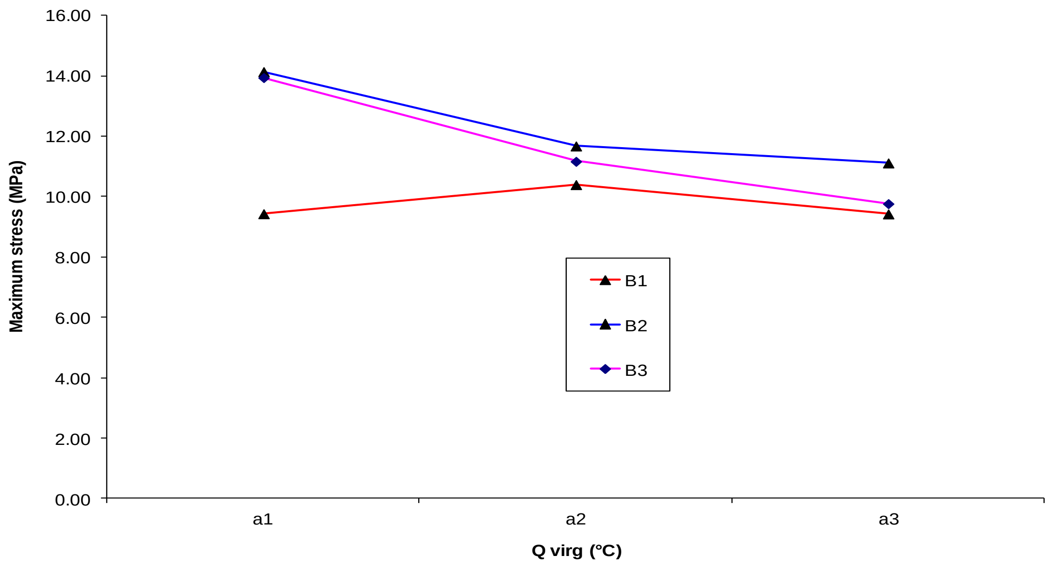


Figure 27 - Iteration between virgin debit and recycled content variables

Consequently another interaction had been proposed to be evaluated, being those interactions, recycled content (b) and calendars rollers temperature (c) (figure 28). Tending into account properties which can be influenced by these parameters , then it may be concluded that a quite few number of interactions should be observed. Four interactions can be observed which indicates that implementing more recycled material and increasing cooling temperature, tensile results may be influenced. Those parameters change the degree of crystalinity since an increament in calendars temperature provides further time to cool and promote the formation of more nucleus which increases capacity to support tensile requests . By other side, in the same way, increasing recycled content, means that polypropylene presented in the blend increases, as results crystalline zones increases as a function of the increament in calendars rollers temperature.

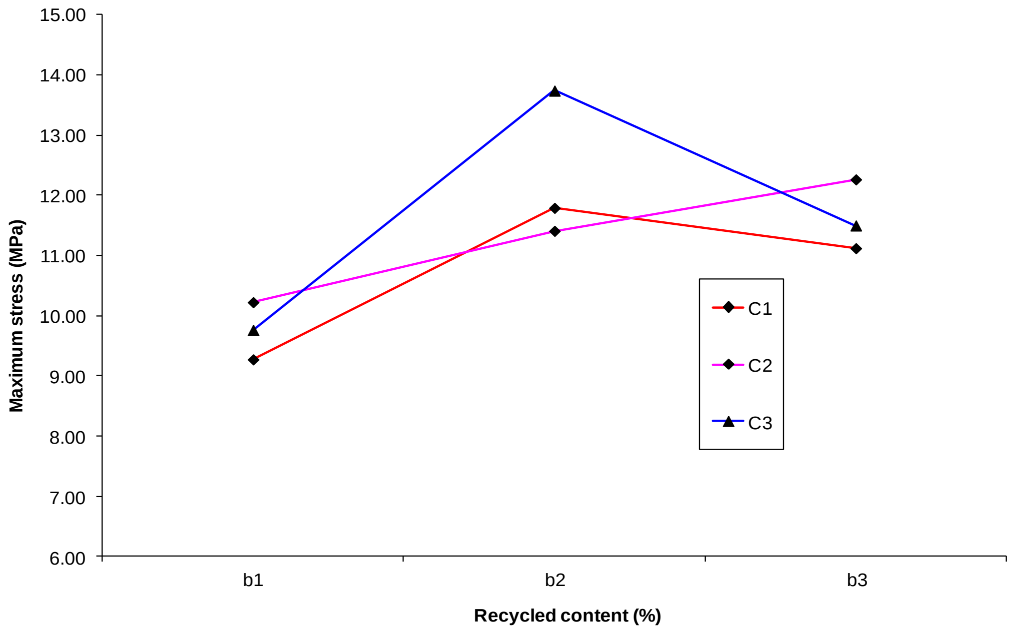


Figure 28 - Iteration between recycled content and rolls temperature variables

Consecutively, the influence of the factors levels tending in consideration the numerical model used for this plan of experiences, analyzing the standard deviation might be proceeded.

Results may be also explained by the statistical interpretation. Tending into consideration of these words, this interpretation should be applied to improve final conclusions of this work (Table 15). In a nutshell, our sample of population is all the samples subjected to the tensile experiment separated by 10 samples which have been included six parameters and two degrees of freedom to be studied. Statistically, some parameters do not contain any influence to the mechanical properties , as a quite few number of examples found, there are rolls temperature (c), tear ratio (f) and interaction A (between the variables a and b). In fact, probably of these parameters to contain influence to the tensile stress have been studied and consequently available, where the probability to influence samples behaviour in one interval 1-α , where α is 5% or 1%. Taking into consideration results on Table 15, variables c, f and interaction B may not be considered as preponderant variables, since practical value is below of the theoretical values.

Table 15 - Statistical results

Sources	F calculated	F table 5%	F table 1%	Standard deviation
a	11.12	4.26	8.02	0.53
b	13.37	4.26	8.02	0.53
c	1.70	4.26	8.02	0.53
d	4.66	4.26	8.02	0.53
e	10.80	4.26	8.02	0.53
f	2.96	4.26	8.02	0.53
A	3.86	3.63	6.42	0.57
B	1.85	3.63	6.42	0.57

In the figure 29 are plotted the contribution of each variable to the improvement of the mechanical properties. These values were obtained from the numerical model.

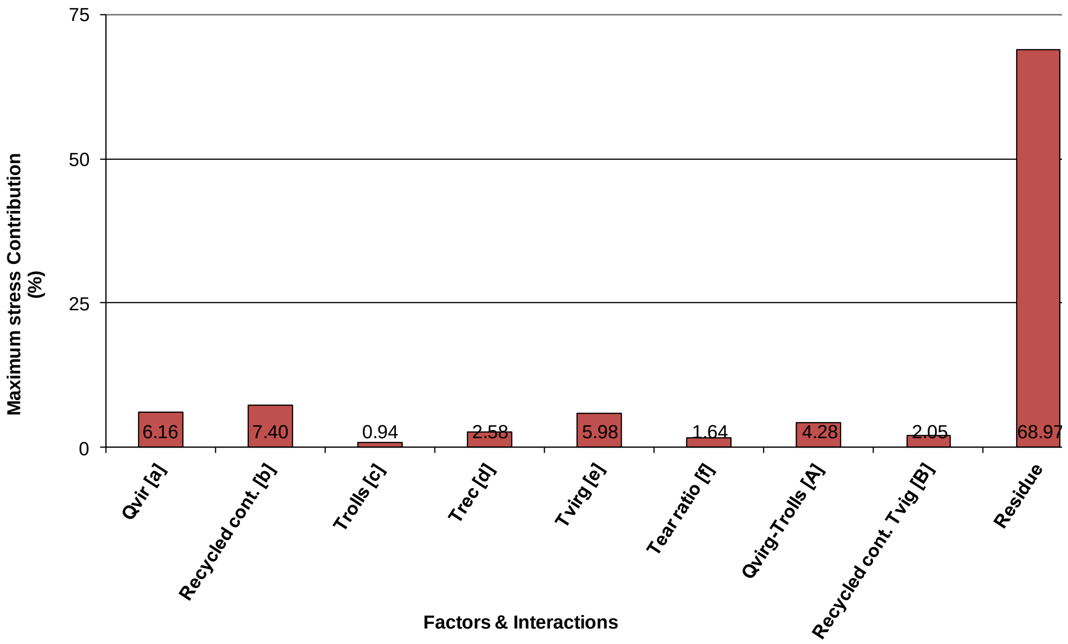


Figure 29 - Contribution of the variables graph -

In this case, levels which allows improving maximum stress in the longitudinal direction have been a1, b2, d1, e1 and interaction A.

The study of the effect of the processing conditions for the maximum stress in the transversal direction had been made and the results are represented in the figure 30.

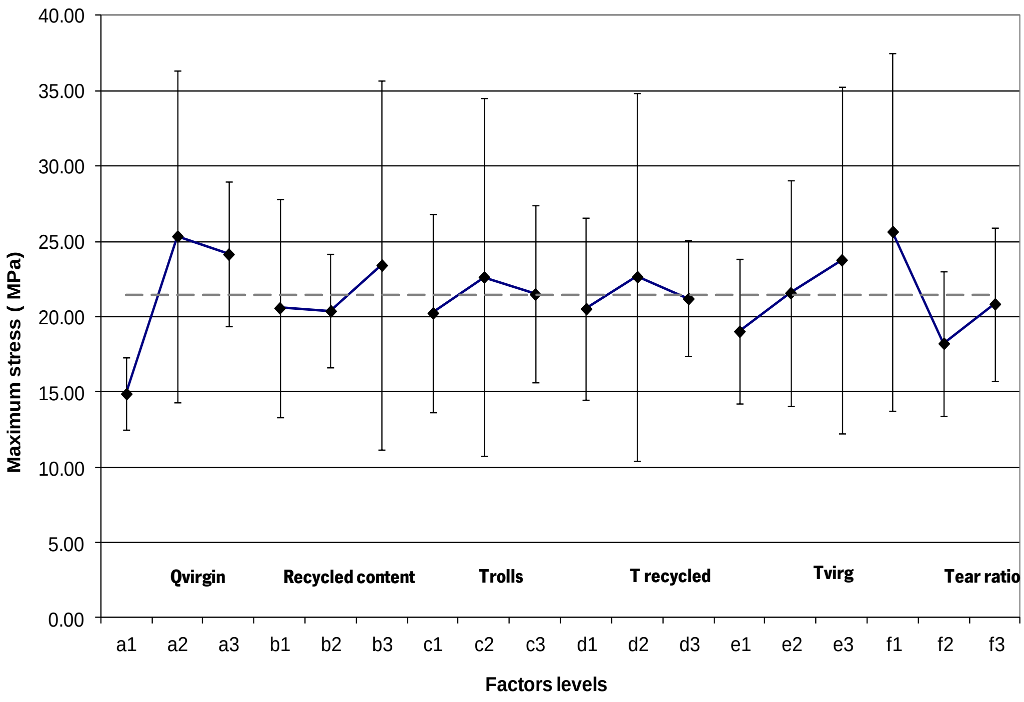


Figure 30 - Effect of variables in the maximum stress for the transversal direction

In this case the confidence interval is: $CI = (25{,}66 - 14{,}90) = 10{,}7\%$. The result obtained is 10,7%, meaning that the results can be accepted.

Analysing the results (Figure 30), it is observed that the variable F has a negative effect, the variables a, b and e have a positive effect and the variables c and d own a neutral effect on the tensile stress at maxim force. The variable f in lowest level, the variables b and e in the higher level and the variables a in the medium level have more contribution for have a higher maximum stress. These results can be explained based on the theoretical concepts referred in the literature subchapter.

The debit of the virgin material (a) has great influence on the maximum stress , about 6,09%. Nevertheless, it occurs in the medium level, since the debit in virgin material which have been implemented was in the medium value, as a consequence the maximum stress and maximum stress reached have been greater. In the transversal direction, thickness may appears as a preponderant factor to increase maximum stress, however as it have been proved, a instability between these three flows are obviously present which origin poor optical and adhesion properties between layers at the exit of the die. Submitting samples to the mechanical requests, a slipping between layers will appears, demonstrating several instability flow during extrusion process. Nevertheless, applying more material to the layers may difficult this slip, resulting in a small improvement of maximum stress.

The content in recycled material (b) contains a positive influence to the improvement in maximum stress for the transversal direction. It allows understanding that an increasing in recycled content promotes an increase in thickness which are responsible to the increasing in maximum stress. Interpretation may be similar, such as, virgin material debit.

Temperature of the virgin LDPE owns a positive influence to the maximum stress in the transversal direction value, about 1,05. As recycled material possess a higher viscosity than virgin material, encapsulation by this material may occur. Then increasing temperature to the virgin material allows increasing much more viscosity difference, as a consequence virgin material may not blend with recycled material. Therefore, a interfacial region of separation may be definitely observed which will improves mechanical properties.

The tear ration (f), has a considerable influence on the maximum stress with a percentage of 2,64%, when it have been in the medium level. When the tear ratio is high, as result the maximum stress is lowest. The orientation of the molecules is frozen by the fast cooling then it allows increasing modulus as a function of tear ratio. An increasing in tear ratio promotes much more orientation which allows decreasing capacity to support in the transversal direction since molecular orientation promoted by the puller rollers contains greater importance to the longitudinal direction. Having into account, intramolecular and intermolecular forces, first one is further difficult to destroy then as in the transversal direction intermolecular forces are preferentially attacked , an decreasing in maximum stress occurs as a function of much more orientation in the longitudinal direction.

The temperature of the calendars rollers (c) has approximately an neutral influence on the maximum stress, about 0,54%. Moreover, statically 0,54% does not contain great importance. The temperature of the calendar rollers have a direct effect on the material structure, tending into consideration that temperature applied have been expanded, an increasement of the degree of crystalinity might be observed and occurred, which may promote much more strength resistance. Yet as materials implemented contains a high difference in viscosity, this influence may not be observed such in a case using materials containing less difference in viscosity.

Temperature of the recycled material (d) owns a neutral role to the tensile results in the transversal direction, which may be explained by the rheological properties of this material. As this material contains a higher viscosity than virgin material, an increase in 30°C in temperature does not allow increase capacity to improve interfacial stability, therefore may it not influence mechanical properties.

The effect of the other variables on the maximum tensile stress is about 10,8% and for that reason the influence can be considered important. Yet, residual influence is too high. Interaction between factors will be evaluated next Figure 31.

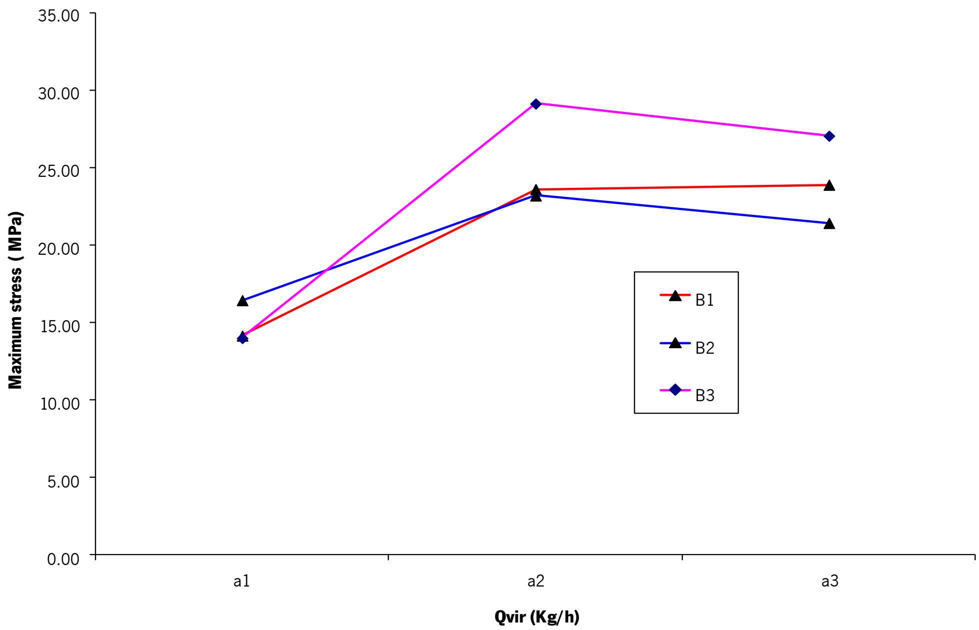


Figure 31 - Iteration between virgin debit and recycled content variables

Three interaction are represented in the figure 31. It takes place when debits are employed in lowest levels. In these levels, material extruded is lowest, as a result the thickness of the sheets decreases. A decreasing in thickness allows decreasing difficulty to promote homogeneity in terms of thickness. As a consequence, sheets can be more prepared to support tensile forces in the transversal direction.

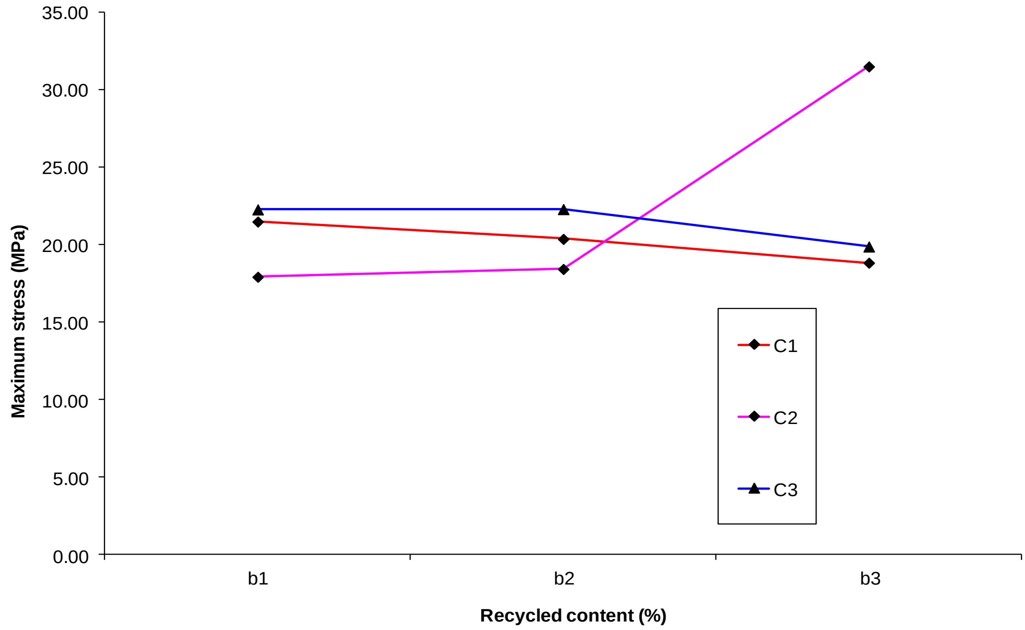


Figure 32 - Iteration between recycled content and rolls temperature variables

Two interactions are perfectly observed (Figure 32). It occurs when recycled debit is used between 50% and 75% and virgin material extrusion temperature is 185°C. Nevertheless, it does not contains sense since behaviour have been completely different and misaligned tending into account other levels present on this graph. A quite few number of tensile results have had an unstable bahaviour, existing a difference of 50 N in force between samples results. This appears to be the better explanation to these results.

Following, tensile results tending into account statistical theory and practical results may be explained (Table 16). Taking into consideration results on table 16, variables b, c, f, e and interaction A and B may not be considered as preponderant variables, since practical value is below of the theoretical results using F Snedecor as a function of dispersion. These results have been obtained because standard deviation is quite large, then future results may not be previewed. This results took into account the analytical model referenced on the DOE presentation.

Table 16 – Statistical results

Sources	F calculated	F table 5%	F table 1%	Standard deviation
a	8.92	4.26	8.02	0.53
b	0.79	4.26	8.02	0.53
c	0.39	4.26	8.02	0.53
d	0.32	4.26	8.02	0.53
e	1.53	4.26	8.02	0.53
f	3.86	4.26	8.02	0.53
A	0.55	3.63	6.42	0.57
B	2.46	3.63	6.42	0.57

Figure 33 allows understanding contribution of each variable to the results of the maximum stress in the transversal direction.

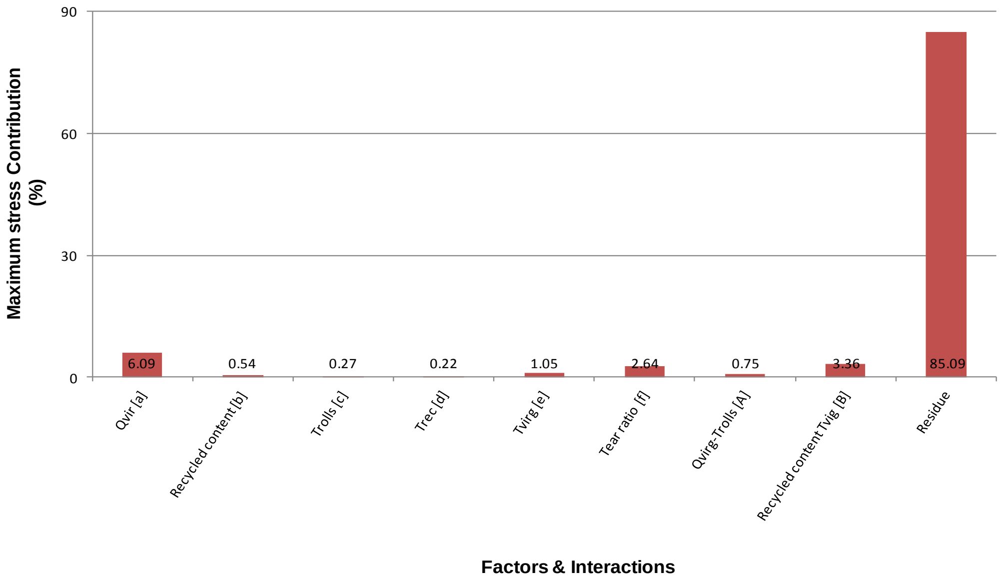


Figure 33 - Contribution graph

In this case, levels which allows improving maximum tensile stress in the transversal direction are a2 , f1 and interaction B.

The effect of the other variables on the tear strength has been measured and consequently plotted (Figure 34) to be evaluate.

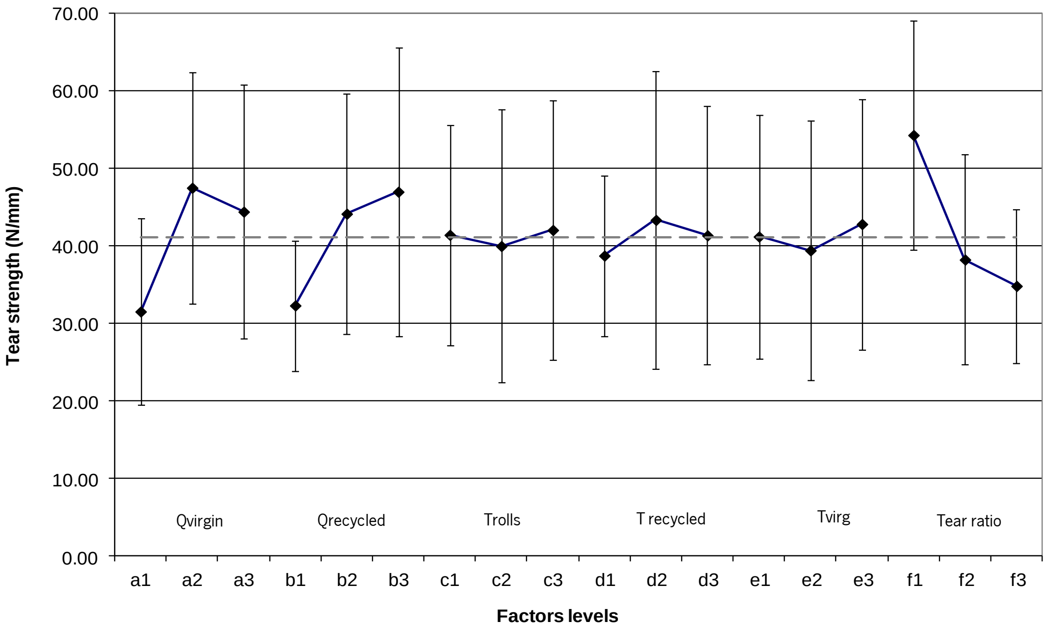


Figure 34 - Tearing strength results

In this case the confidence interval is: $CI = (54{,}36 - 31{,}6) = 23\%$. The result obtained is 23%, meaning that the results can be accepted.

It has been observed that the variable f has a negative effect for the tear strength of the extruded sheet, whereas a and b have a positive effect on it, in relation with e, c and d, a neutral effect is clearly observed. Therefore, the variables f and b in the highest level and the variable a in the medium level have been parameters which contain much more importance to define the tear strength.

The content of the recycled material (b) has a great influence on the tear strength, representing a change of 5,06 %. When recycled content is high, it requires more force to tear the sheet than in the case where 25% or 50% in recycled material is being used. As a conclusion, this is related with the increasing in thickness of the sheet, as result of increase extrusion content in recycled material. Then, as a consequence tear strength is improved.

The debit in virgin material also owns a important influence to improve the mechanical response of the samples in tear strength, and it represents statically 6%. As it was discussed about the increasing in recycled material content, in this case also thickness contains a preponderant role for the improvement in tear strength. The thickness suffer an increment as a function of the increasing in virgin material debit. It will be proved in the microscopic experiments.

The tear ratio (f) contains also a great influence on the tear strength and statically it represents 9,25%. When the tear ratio is highest, samples have more orientation in the flow direction. However , the tear strength is lower in this case because it is related to the high orientation of the molecules, promoting facilities to tear the sheets in this direction. Increase tear ratio also allows decreasing tear strength as results of the thickness loss.

The rolls temperature (c) has neutral influence for the tear strength, and this value have been about 0,09%. The rolls temperature participates to the crystallization of the matrix. Then, results demonstrate that crystallization does not contain influence for the tear strength results. Yet, exists an important aspect, tear strength have been higher when 20 °C and 40 °C in extrusion temperature were used which can be related to the cooling time. A low temperature does not allow growing up crystals, which means that a high temperature allows increasing number of crystals. Eventually, as conclusion a high degree of crystallinity decreases tear strength because of the high level in crystalline cores and using a lowest level by the opposite.

The extrusion temperature of the recycled material includes also a neutral influence on the tear strength, about 0,24%. A high (200 °C) and low (170 °C) temperatures in the extrusion of the recycled material decreases tear strength. On the other side, using 195 °C as extrusion temperature, tear strength is improved. This may result in the presence in content of polypropylene. These material possess different processing and melting temperatures, then it affects stability of the extrusion process and melting homogeneity.

Lastly, extrusion temperature of the virgin material which contains a neutral effect on the tear strength, about 0,24%. However, a inversed influence is observed in terms of the levels effects.

In the following figures, interactions of the variables are plotted.

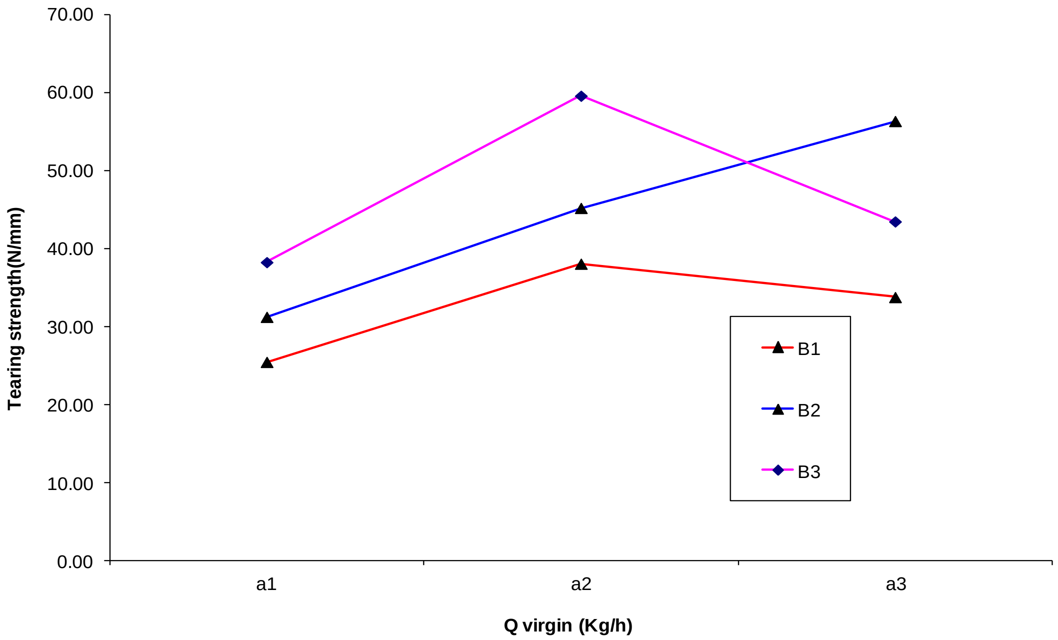


Figure 35 - Iteration between variables A/B (longitudinal direction)

Interaction between virgin debit and recycled content in the extrusion process has been studied. Results are plotted in the Figure 35. Between virgin and recycled content one interaction has been observed, tending into account their influence to the tear strength. Then, this interaction takes place when virgin and recycled materials are extruded in highest levels of debit and content. Interaction present occurs when virgin material is extruded in highest quantity (3,8 Kg/h) and recycled material is extruded using the second and third level of content (50% and 75% respectively). It can be explained by the processing temperature which in highest level allows increasing recycled material viscosity caused by the presence in polypropylene, which as a consequence, homogeneity is improved. As it has been studied in literature survey, these two materials cannot be blended since they are incompatible. Interactions observed might be explained by the viscosity and homogeneity between layers such as it has been explained tending into account virgin and recycled interactions effect.

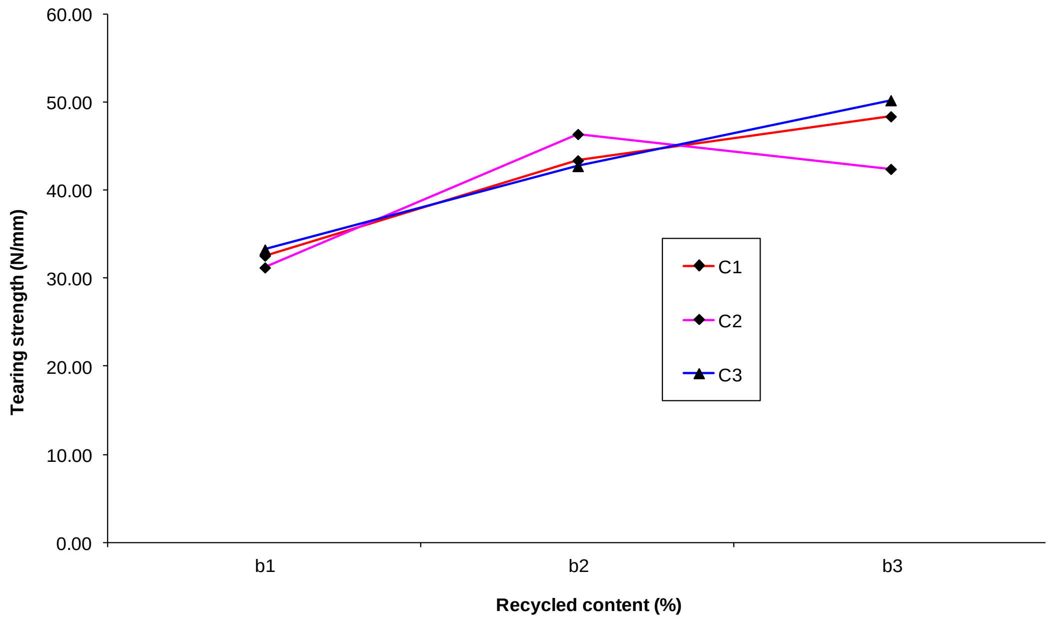


Figure 36 - Iteration between recycled content and rolls temperature

In this situation, interaction between recycled content and calendars rolls temperature in the extrusion have been studied. Results are plotted in the Figure 36. Three interaction have been observed, tending into account their influence to the tear strength. As a preliminary remark, these interactions occurs when recycled material had been extruded in all levels of contents and calendars rollers temperatures. These interaction may be explained by the influence of the calendars rollers to the cooling and crystallization of the sheet. As a conclusion, these factors depends on each other to the results in tear strength. Calendars rollers has a preponderant role in the degree of crystalinity which applying more recycled material may increase it, tending into consideration that PP is presented in the recycled material.

Following, the tearing experiments results, tending into account statistical theory and practical results from the table 17 may be explained .Taking into consideration results on the table 17, variables c d, e and interactions A and B may not be considered as preponderant variables, since practical value is below of the theoretical result.

Table 17 – Statistical results related to the tearing experiments

Sources	F calculated	F table 5%	F table 1%	Standard deviation
a	9.90	4.26	8.02	0.53
b	8.36	4.26	8.02	0.53
c	0.16	4.26	8.02	0.53
d	0.74	4.26	8.02	0.53
e	0.40	4.26	8.02	0.53
f	15.28	4.26	8.02	0.53
A	2.48	3.63	6.42	0.57
B	0.42	3.63	6.42	0.57

Following Figure 37 where the contributions obtained of each variable to the tearing results, it is demonstrated that residue contains a high importance, however it is not interesting for the results.

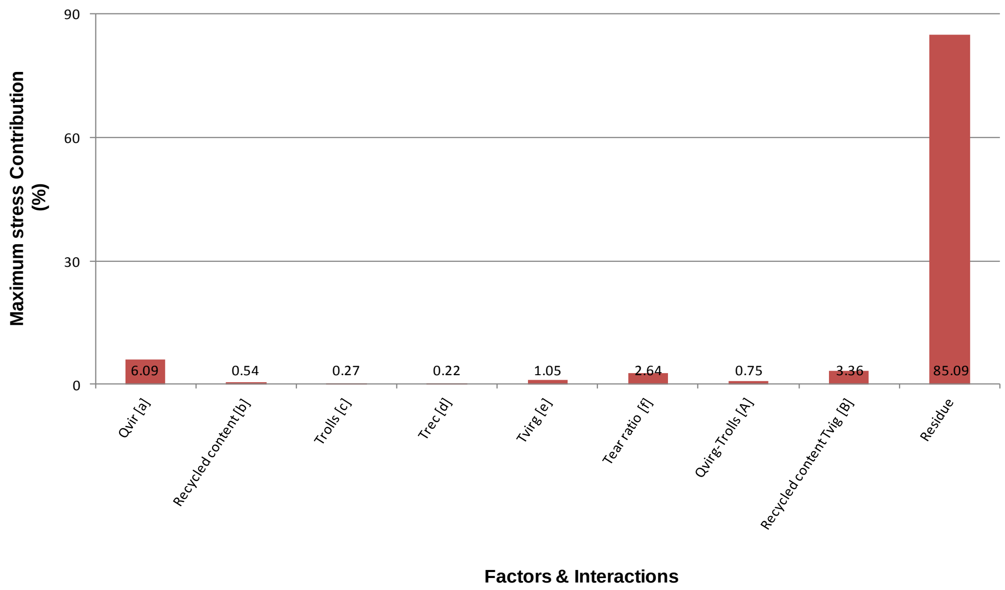


Figure 37 - Contribution graph of the parameters for the tearing results

In this case, levels which allows improving maximum tensile stress in the longitudinal direction have been a2, b3 and f1.

The effect of the other variables on the tearing propagation behaviour was measured and plotted in the Figure 38.

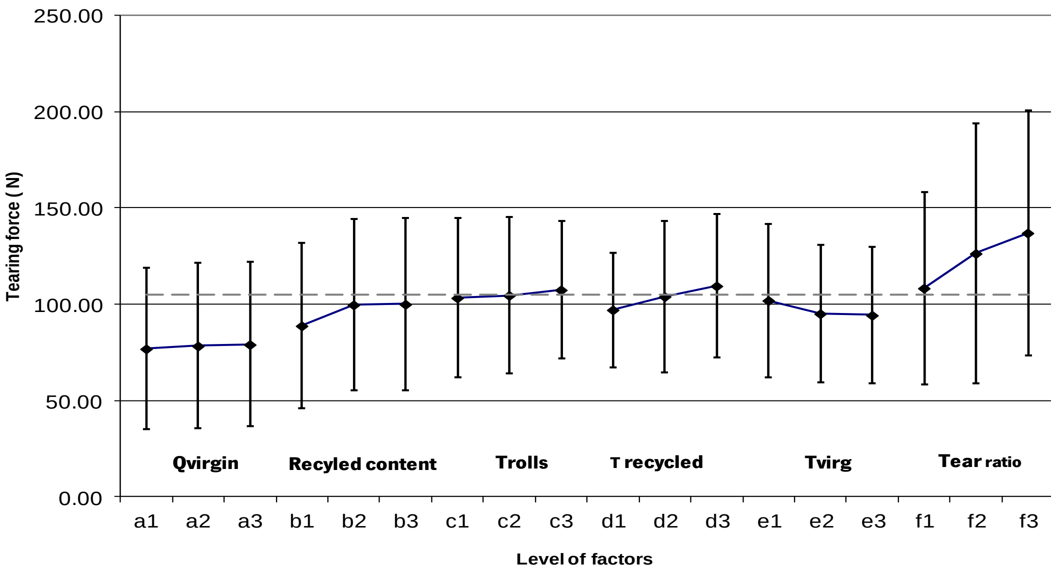


Figure 38 - Effect of variables for the tear propagation behaviour

In this case the confidence interval is: $CI = (140 - 80) = 60\%$. The result obtained is 60%, meaning that the results cannot be totally accepted.

Analyzing the results in the Figure 38, it has been observed that the variable e has a negative effect, the variables b, c, d and F have a positive effect and the variable a own a neutral effect on the strength measurements to the tearing propagation. The variables f and c in lowest level, the variable b, a and d in the highest level and the variables e in the medium level contain more contribution to reach a higher strength. These results can be explained based on the theoretical concepts referred in the literature chapter.

The debit of the virgin material (a) has great influence on the maximum force 28,95%, but it occurred in the highest level. When the debit in virgin material is higher, maximum force reached have been greater which it may be related to the thickness since it suffers one increasing, requiring more energy to tear.

The content in recycled material (b) contains a positive influence to the improvement in maximum strength to the tearing propagation, about 1,89%. It allows understanding that an increasing in recycled content promotes an increase in thickness which may be responsible for this improvement. As a conclusion, an increase of thickness is regarded as the responsible aspect to the strength improvement.

Temperature of the virgin LDPE owns a neutral influence to the maximum strength to the tearing propagation. However, in the lower level , strength have been somewhat higher.

Calendars rolls temperature has a great influence on the strength to the tearing propagation, about 5,12%. Manly, this variable affects particularly the degree of crystallization, as result maximum strength increases with the increasing of the rolls temperature, where an increaseof the degree of crystalinity may be observed.

The tear ration, (f), has a considerable influence on the maximum stress with a percentage of 24,93%, when it has been in a lowest level. When the tear ratio is high the maximum force is lower. The orientation of the molecules is frozen by the fast cooling then it allows decreasing tear strength as a function of tear ratio. Increasing tear ratio, the thickness suffer a decrease and molecular orientation increases, as a result force required to tear is slightest.

The temperature of the recycled material has had an important influence on the maximum stress, about 4,4%. The temperature of the recycled material includes a direct effect on the material structure, since if the recycled temperature is increases, degree of crystalinity might be increased, which it promotes much more strength. Moreover, its related to the PP presented in the blend as well.

The effect of the other variables on the maximum strength to the tearing propagation is about 14%, then the influence of these parameters can be considered as important. Though, residual effect have been narrow which is good.

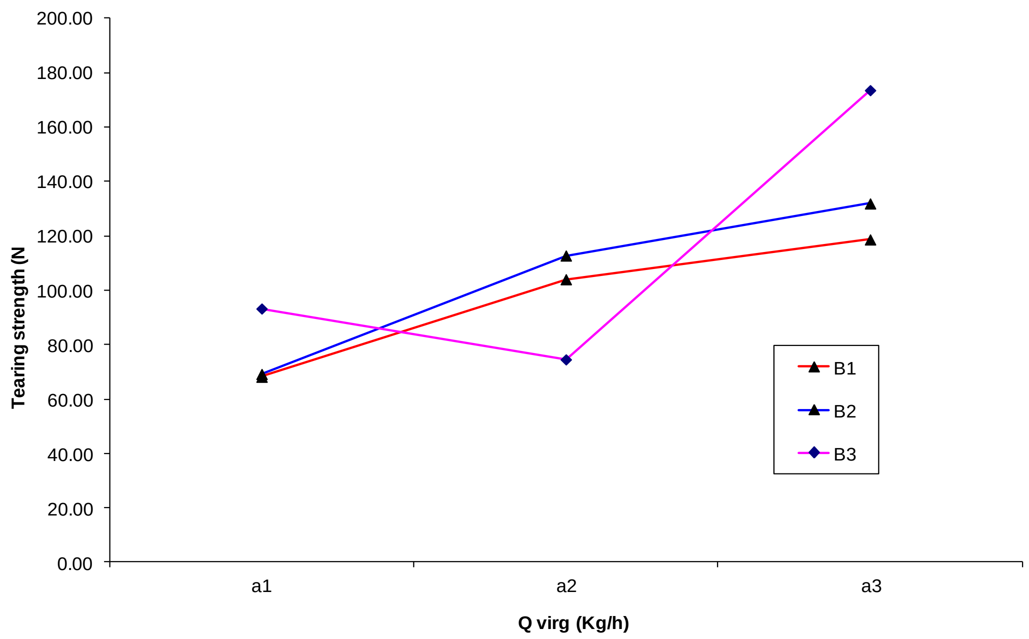


Figure 39 - Interaction between virgin debit and recycled content

Four interactions can be observed tending into account virgin debit and recycled content (Figure 39). As these parameters allow increasing thickness, which may improve tearing strength, then it is normal to observe these interactions.

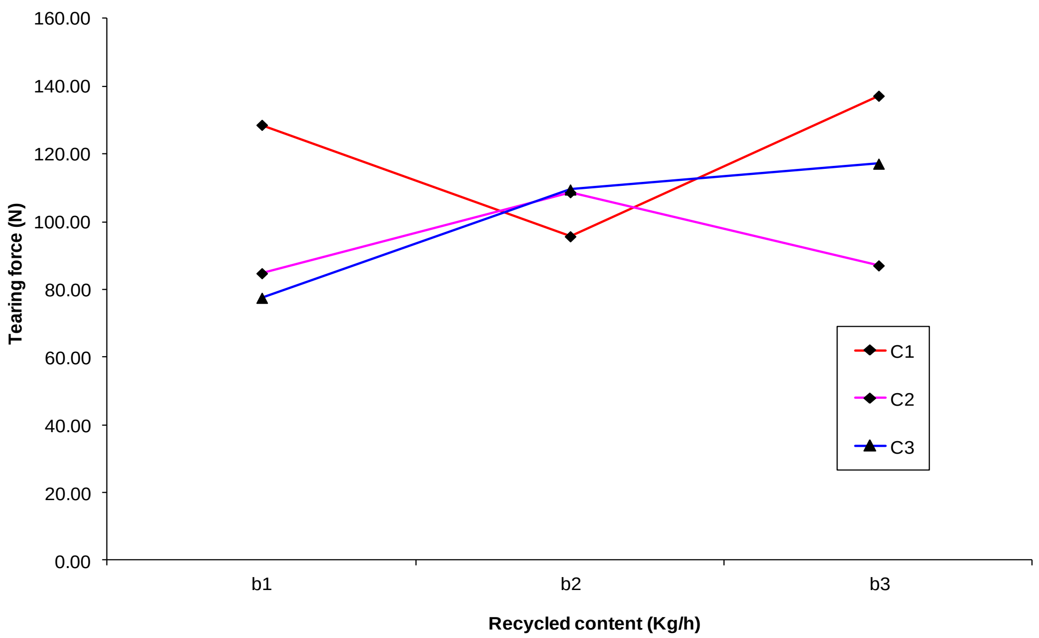


Figure 40 – Interaction between recycled content and calendars temperature

In this case (Figure 40), other four interaction are observed, such as, in the previous case. In this situation, degree of crystalinity might be related to the curves behaviour in this interaction, since an increase in degree of crystalinity allows a decrease on the capacity to support tear forces, tending into consideration that molecules are more oriented which facilities tear propagation between molecules.

Following tearing experiments results tending into account statistical theory and practical results may be explained (Table 18). Taking into consideration results on the table 18, all variables may be considered as preponderant, since practical value is above of the theoretical result. As standard deviation of the results in this case is lower, then results can be previewed as expected to be inside of this population in the next experiments.

Table 18 – Statistical results related to propagation behaviour

Sources	F calculated	F table 5%	F table 1%	Standard deviation
a	445.72	4.26	8.02	0.53
b	29.11	4.26	8.02	0.53
c	78.77	4.26	8.02	0.53
d	63.69	4.26	8.02	0.53
e	163.50	4.26	8.02	0.53
f	383.79	4.26	8.02	0.53
A	82.58	3.63	6.42	0.57
B	60.26	3.63	6.42	0.57

Following in the figure 41 is available contributions obtained of each variable to the tearing results, where is demonstrated that residue contains a high importance which is not interesting for the results.

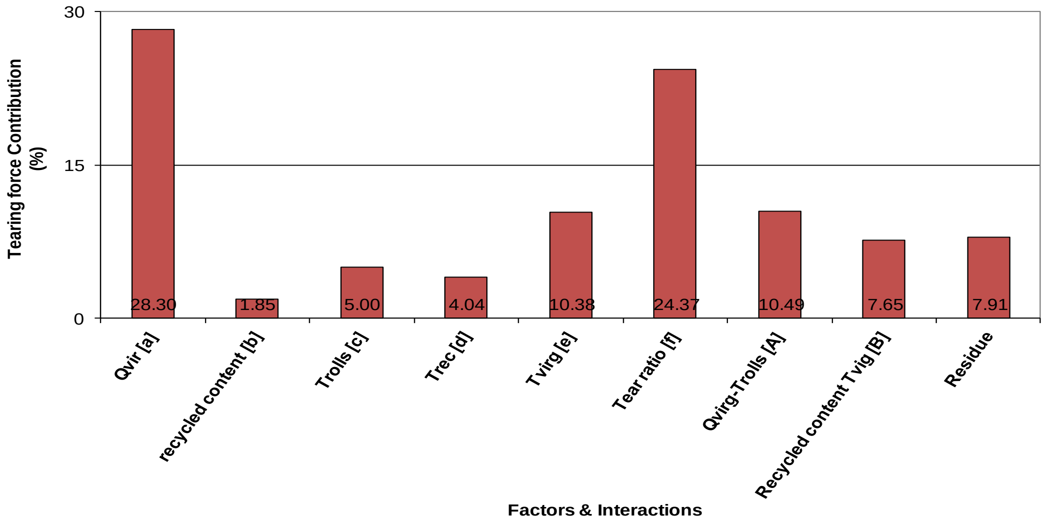
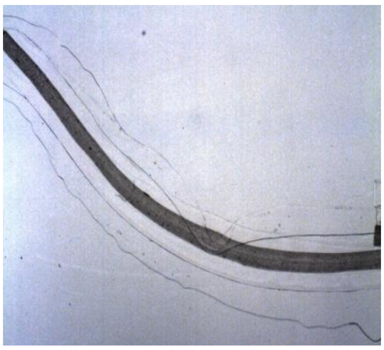


Figure 41 - Contribution of the variables to the results

6. MICROSCOPIC ANALYSIS RESULTS

To better understand the thickness present on the layer containing recycled material, bright field microscopy was applied to observe some samples. A constant thickness or not can be decisive to the mechanical behaviour of the samples. If the thickness of the central layer containing recycled material is not constant, then it can be supposed that the layers had a bad contact during the exit in the die of the extrusion machine. It can be explained tending in consideration the incompatibility existent between the polyolefins, (PP and PE) present in the recycled material proved by the DSC.



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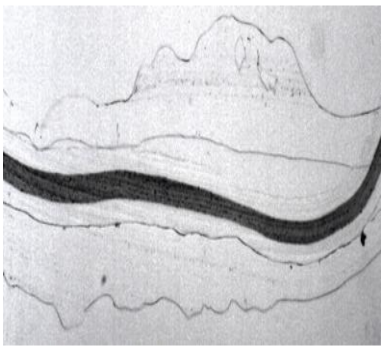

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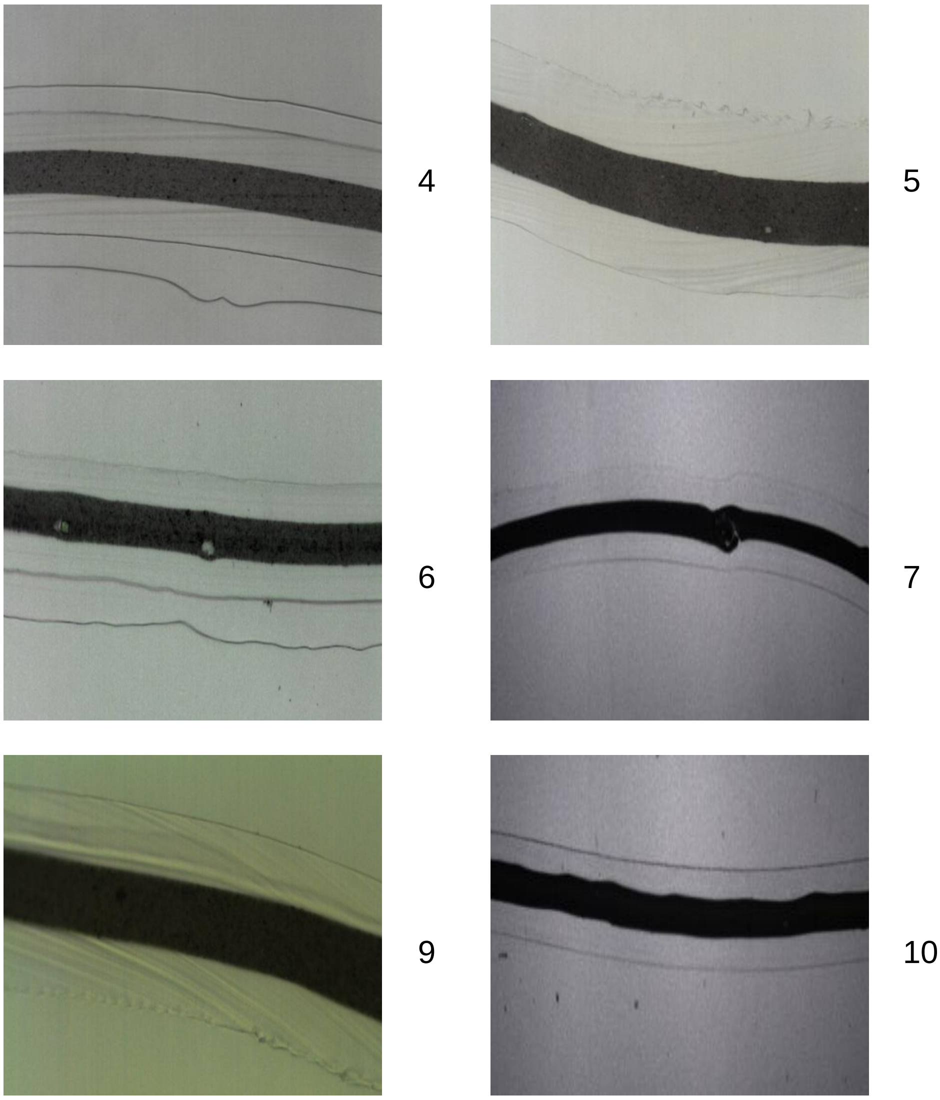

Figure 42 – Thickness details of the 2, 3, 4, 5, 6, 7, 9 and 10 samples

The figure 42 represent some of these results and it can be observed an inconstant thickness along the width. It can be used to explain some results obtained by the mechanical experiments. As conclusion, the debit control performed to ensure the layer's thickness and adhesion was not reached.

7. Conclusions

Faced with a common problem, which aims to determine the processing conditions that enhance the mechanical performance of one material, it was applied the DOE analysis, in order to, with only a few experiments and a short amount of material, obtain a credible solution.

Implementing DOE technique have been determined the optimal set of factors, with which, have been obtained the best mechanical responses to the experiments solicited. It should be made a critical analysis to the results, in order to reach how far it might be a solution or not, and then accept it as a plausible solution or not.

The optimal set of factors obtained, are at all according with the expected, so this was a positive indicator that all have been proceeded as planned.

For the tensile experiments, it has been found that the tear ratio, virgin material debit, recycled content and virgin material have been the variables most influents. In regard to the tearing experiment, it has been observed that tear ratio, recycled content and virgin debit have been the variables that contains greater influence, as could be expected, since it influence thickness. Such affirmation have been proved by the last experiment that has been the study of the strength to the tearing propagation.

It was decided not to consider the change of the variables values in which the interference in the response was zero or close to that. This decision may be a possible explication to something that is incorrect as this change could have made a small difference, but essential in the final results.

There are some important aspects that should be considered such as this plan of experiments which is restrictedly available to these three intervals or temperatures levels, tear ratio and debits. Outside of these values, sheet behaviour may be completely different and it cannot be previewed. A new method to separate materials in the previous stage to the recycling of the materials has been proposed to eliminate the presence of the PP, another development should be to use compatibilizers.

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APPENDIXES

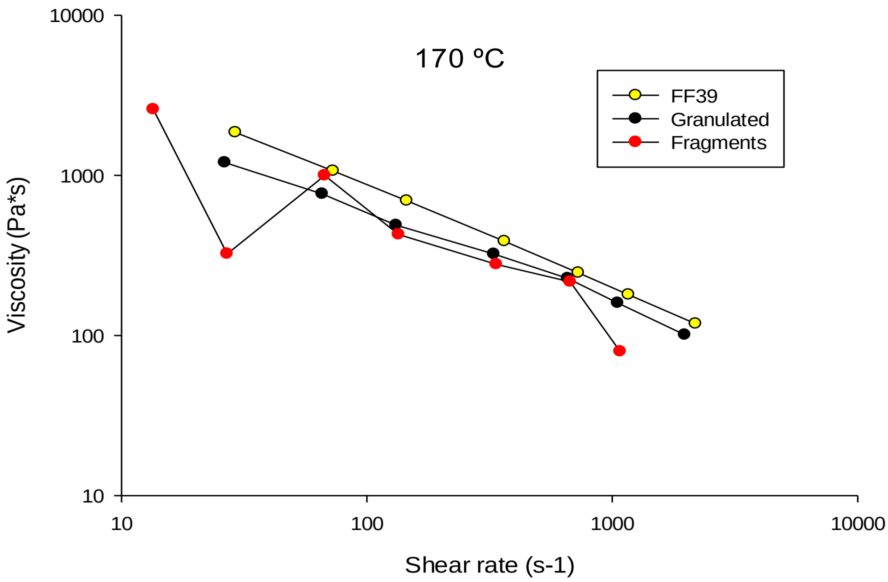

Figure 43 - Rheological curves of the materials to 170°C

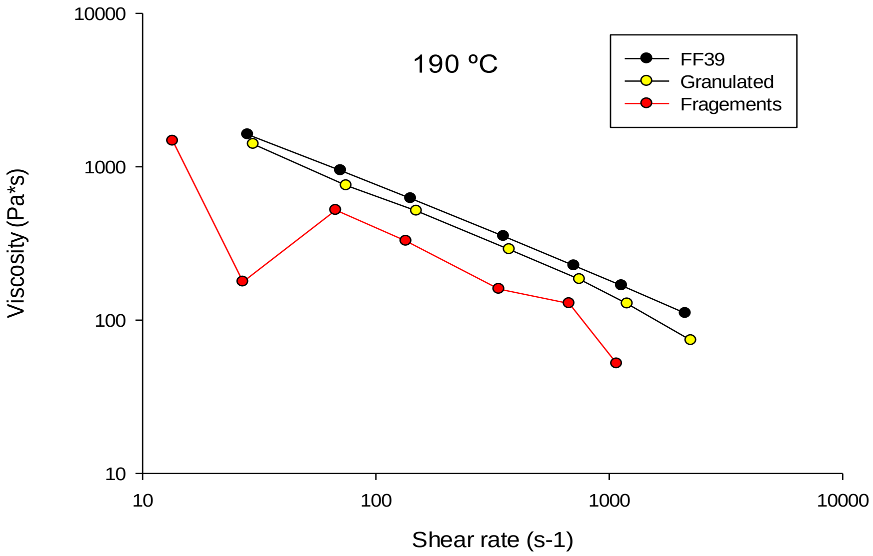

Figure 44 - Rheological curves of the materials to 190°C

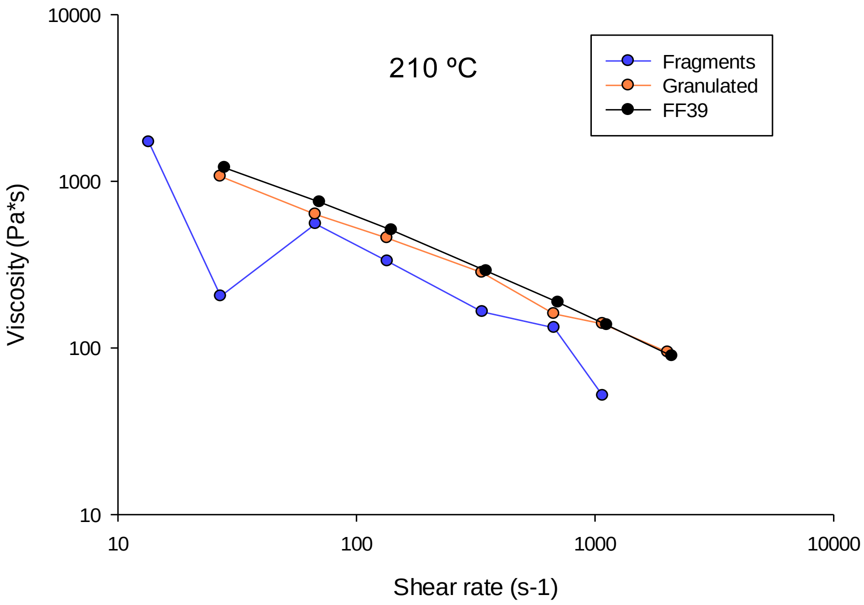

Figure 45 - Rheological curves of the material to 210°C

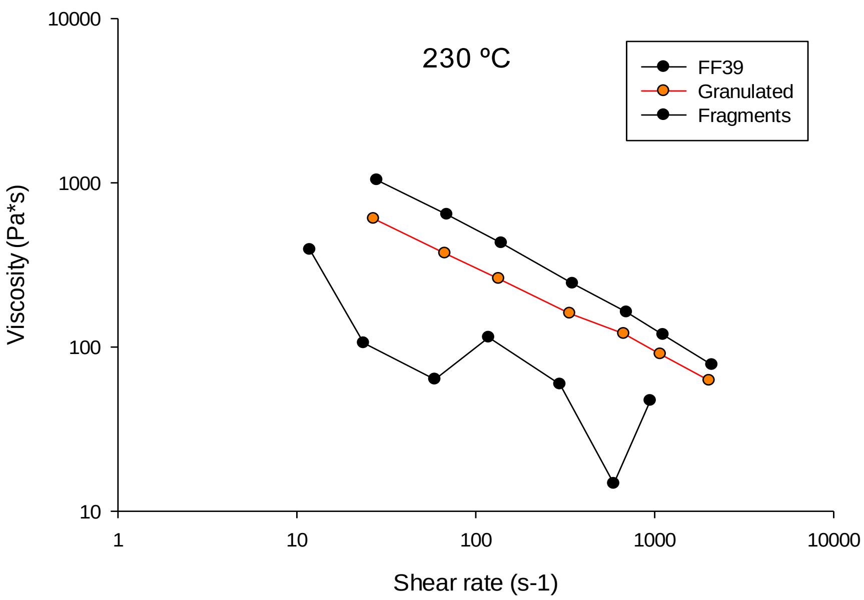

Figure 46 - Rheological curves of the material to 230°C

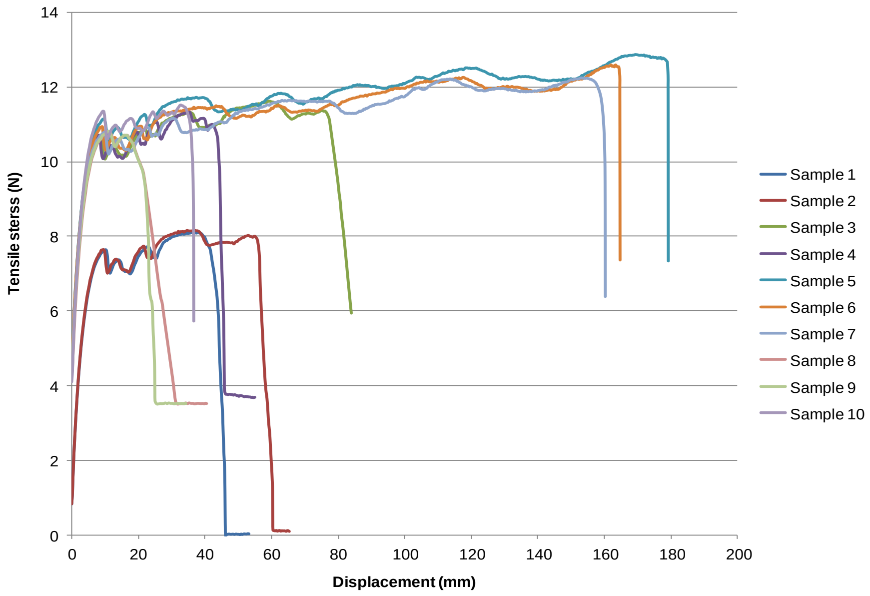

Figure 47 - Tensile tests L1 in the longitudinal direction

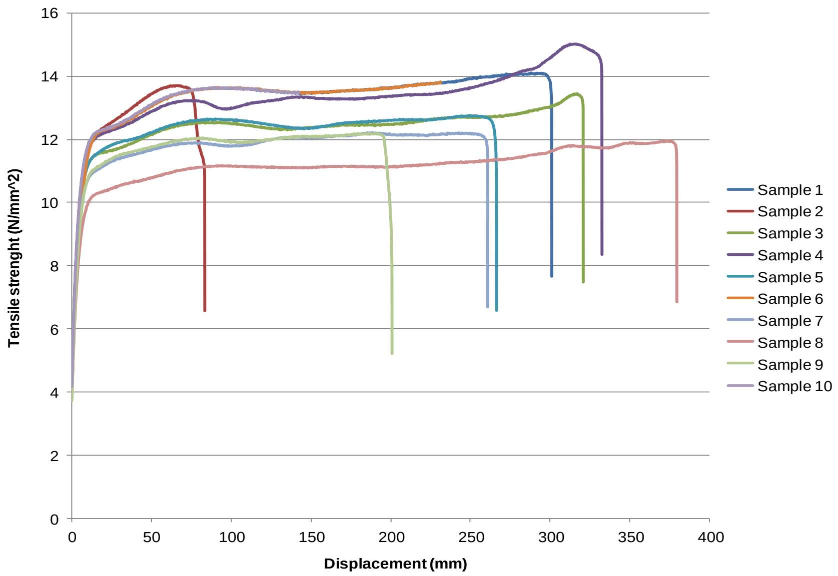

Figure 48 - Tensile tests L1 in the transversal direction

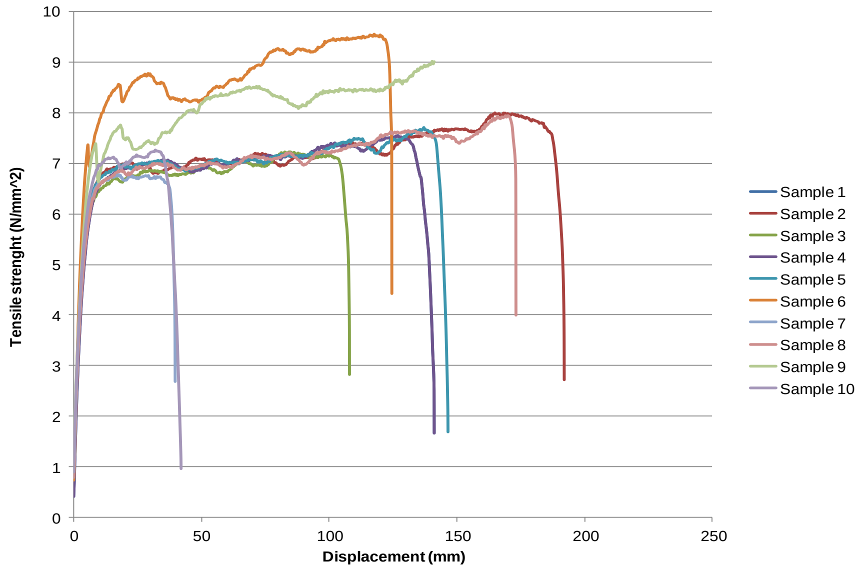

Figure 49 - Tensile results L2 in the longitudinal direction

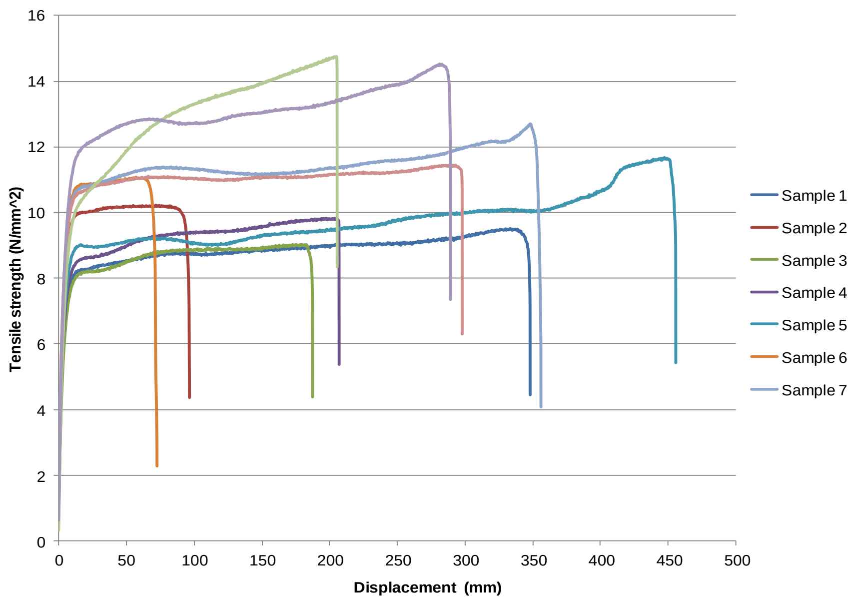

Figure 50 - Tensile results L2 in the transversal direction

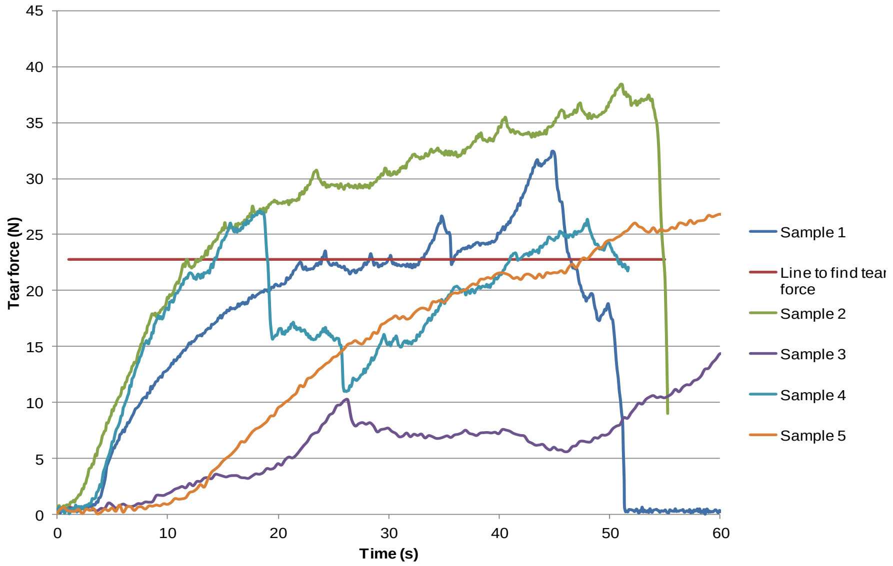

Figure 51 - Tearing test results L1 transversal

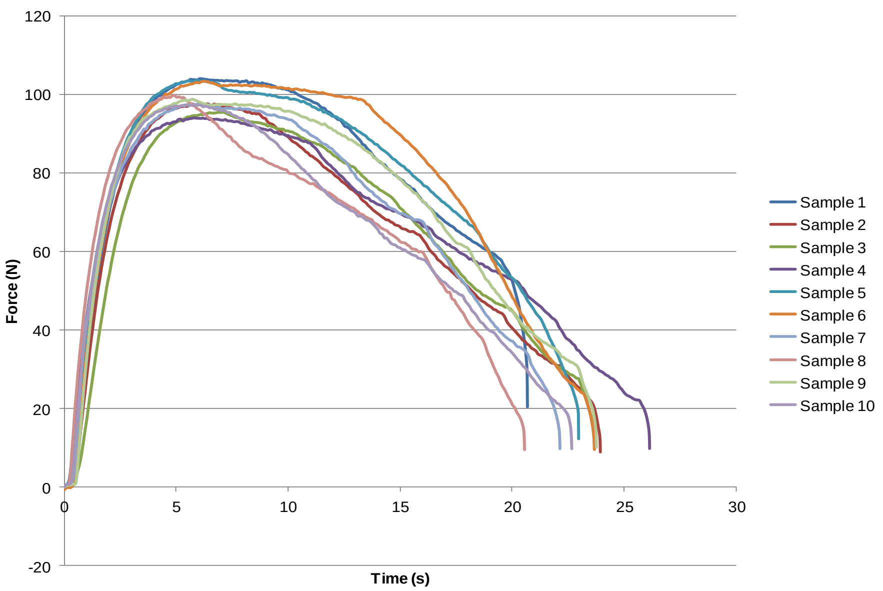

Figure 52 - Strength behaviour to the tear propagation

Polimeri Europa

Blown Film Resins

Technical Data Sheet

Riblene® FF39

Low Density Polyethylene

DESCRIPTION

Riblene® FF39 is a high molecular weight low density polyethylene (LDPE), suitable for blown film extrusion. Riblene® FF39 is characterised by a good melt strength leading to a good bubble stability during extrusion. Films manufactured by Riblene® FF39 are easily heat shrinkable and characterised by good optical and mechanical properties.

APPLICATIONS

Riblene® FF39 is recommended for the production of shrink film for medium and light loads, for dipers and for blends.

PROPERTIES

TYPICAL

Properties		Test Method	Typical value
Resin Properties			
Melt Flow Index$_{190\ °C\ /\ 2.16\ kg}$ (g/10')		ASTM D1238	0.7
Density (g/cm^3)		ASTM D1505	0,924
Melting point (°C)		PE	114
Brittleness point (°C)		ASTM D746	<-75
Vicat softening point (°C)		ASTM D1525	95
Film Properties[1] - Thickness 70 μm			
Tensile strength at yield (MPa)	MD	ASTM D882	11
	TD		12
Tensile strength at break (MPa)	MD	ASTM D882	22
	TD		20
Elongation at break (%)	MD	ASTM D882	400
	TD		600
1% Secant Modulus (MPa)	MD	ASTM D882	180
	TD		190
Tear resistance (Elmendorf) (N/mm)	MD	ASTM D1922	45
	TD		60
Impact resistance, F_{50} (g) (Dart Drop Test)		ASTM D1709	180
Coefficient of Friction (dynamic)		ASTM D1894	>0.5
Haze (%)		ASTM D1003	7
Gloss, 45° (%)		ASTM D2457	68

[1] Typical value for a film extruded with BUR 1:3, thickness 70 μm.
Actual properties are typical and may vary depending upon operating conditions and additive package.

This document is constituted of 2 pages

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Page 1/2
May 2006

Figure 53 – Riblene FF39 Datasheet

NORME FRANÇAISE ENREGISTRÉE	MATIÈRES PLASTIQUES *FEUILLES MINCES* **DÉTERMINATION DE LA RÉSISTANCE A LA PROPAGATION DE LA DÉCHIRURE**	**NF T 54-108** Avril 1971

A la date d'enregistrement de la présente norme, il n'existe pas de Recommandation ISO correspondante.

PRÉAMBULE

La résistance à la déchirure des feuilles minces en matières plastiques est une fonction complexe de leur résistance à la rupture par traction. Elle dépend de la géométrie de l'éprouvette, (en particulier de la présence ou non d'une entaille), et de la vitesse d'essai.

Cette détermination peut avoir pour objet ;

soit la résistance globale pour obtenir la déchirure proprement dite, objet de la norme **NF T 54**-107,

soit la résistance à la propagation d'une déchirure, objet de la présente norme.

1 OBJET ET DOMAINE D'APPLICATION

1.1 La présente norme a pour objet de déterminer la résistance à la propagation d'une déchirure amorcée de feuilles minces de matières plastiques. Cette méthode est applicable aux feuilles obtenues à partir de matériaux souples ou rigides.

1.2 Les résultats obtenus par cette méthode fournissent une information permettant de classer comparativement des échantillons de feuilles en matières plastiques de composition semblable.

Pour certaines de ces matières, les résultats obtenus par cette méthode peuvent ne pas être en accord avec leur résistance à la propagation de la déchirure observée dans la pratique.

1.3 La résistance à la propagation de la déchirure des feuilles minces en matières plastiques dépend en partie de l'épaisseur de l'échantillon, mais elle n'est généralement pas en relation simple avec cette épaisseur. Les résistances mesurées par cette présente méthode ne peuvent pas être considérées comme valables pour une large gamme d'épaisseur, sous peine de donner des résultats erronés par rapport à la véritable résistance à la propagation de la déchirure du matériau. Les résultats fournis par cette méthode ne peuvent être comparés que pour des échantillons dont les épaisseurs ne diffèrent pas de plus de ± 10 % de l'épaisseur nominale ou moyenne de ces échantillons.

Enregistrée par décision du 26-3-71	**Les observations relatives à la présente norme doivent être adressées à l'AFNOR TOUR EUROPE CEDEX 7 92 PARIS-LA DÉFENSE**	

Imp. Chambord NF T 54-108 - 1er Tirage 4-71

Éditée par l'ASSOCIATION FRANÇAISE DE NORMALISATION (AFNOR) - Tour EUROPE - CEDEX 7 - 92 PARIS-LA DÉFENSE - Tél. (1) 788 11-11

Plastics. Thin Sheets. Determination of resistance to tear propagation.

Figure 54 – French norm to the traction experiments

ISSN 0335-3931

norme européenne

norme française

NF EN ISO 527-3
Octobre 1995

Indice de classement : **T 51-034-3**

ICS : 83.140.00

Plastiques

Détermination des propriétés en traction

Partie 3 : Conditions d´essai pour films et feuilles

E : Plastics - Determination of tensile properties -
Part 3 : Test conditions for films and sheets
D : Kunststoffe - Bestimmung der Zugeigenschaften -
Teil 3 : Prüfbedingungen für Folien und Tafeln

Norme française homologuée

par décision du Directeur Général de l´AFNOR le 20 septembre 1995 pour prendre effet le 20 octobre 1995.

Remplace la norme homologuée NF T 51-034, de décembre 1986.

Correspondance La norme européenne EN ISO 527-3:1995 a le statut d´une norme française. Elle reproduit intégralement la norme internationale ISO 527-3:1995.

Analyse Le présent document spécifie les conditions pour la détermination des propriétés en traction des films et feuilles en matières plastiques, de moins de 1 mm d´épaisseur, à l´exception des matériaux cellulaires et des plastiques renforcés de fibres textiles, par la méthode d´essai décrite dans la partie 1.

Descripteurs **Thésaurus International Technique :** plastique, film, feuille plastique, essai, essai de traction, conditions d´essai, appareillage, éprouvette d´essai, allongement à la rupture, contrainte de traction, module d´élasticité, schéma.

Modifications Mise à jour de la norme NF T 51-034 par reprise de la norme ISO 527-3 en norme européenne.

Corrections

Éditée et diffusée par l´Association Française de Normalisation (AFNOR), Tour Europe 92049 Paris La Défense Cedex — Tél. : (1) 42 91 55 55

 1er tirage 95-10

Figure 55 – French norm to the tearing experiment

NORME FRANÇAISE HOMOLOGUEE	PLASTIQUES **FILM ET FEUILLE** **DETERMINATION DE LA RESISTANCE AU DECHIREMENT** **Partie 1 : Méthode de déchirement pantalon**	**NF** **T 54-140** **Octobre 1984** **ISO 6383/1**

éditée par l'association française de normalisation (afnor) – tour europe cédex 7 92080 paris la défense – tél. (1) 778-13-26

AVANT PROPOS

A sa date de publication, la présente norme reproduit la norme internationale ISO 6383/1 – 1983.

Les références aux normes françaises correspondant aux normes ISO citées au chapitre 2 "Références" et dans le cours du texte sont les suivantes :

NFT 51-014 ISO 291	Plastiques – Atmosphères normales de conditionnement et d'essai.
NF T 54-102 ISO 1184	Matières plastiques – Feuilles – Détermination des caractéristiques en traction.
NF T 54-101 ISO 4591	Matières plastiques – Feuilles – Présentation et dimensions – Méthodes de contrôle.
NF T 54-101 ISO 4593	Matières plastiques – Feuilles – Présentation et dimensions – Méthode de contrôle.

Homologuée par décision du 1984-09-05 Effet le 1984-10-05		© afnor 1984 Droits de reproduction et de traduction réservés pour tous pays

Soregraph, impr., 92300 Levallois NF T 54-140 1er TIRAGE 84-09

Plastics - Film and sheeting - Determination of tear resistance - Part 1: Trouser tear method

Kunststoffe - Folien und Bahnen - Bestimmung des Reisswiderstandes - Teil 1 : Schlitzmethode

Figure 56 - French norm to the determination of the strength to the tearing propagation

NF T 54-140 — 6 —

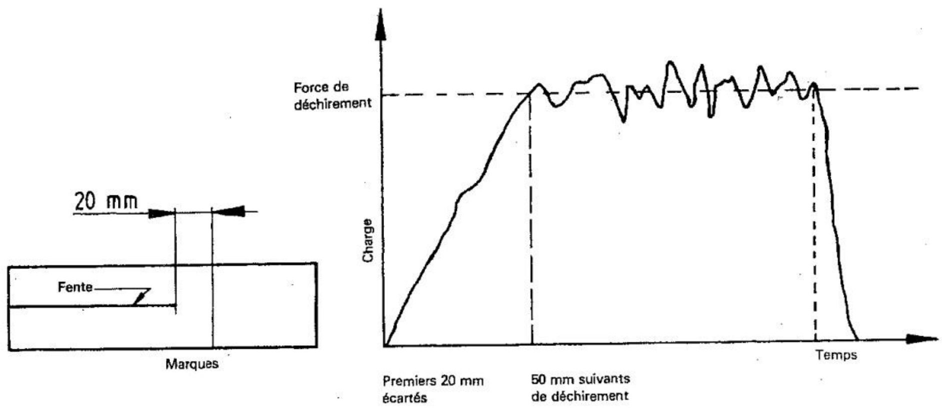

Figure 4 — Graphique charge/temps avec une partie plateau

Figure 57 – Typical curve of the tearing experiment

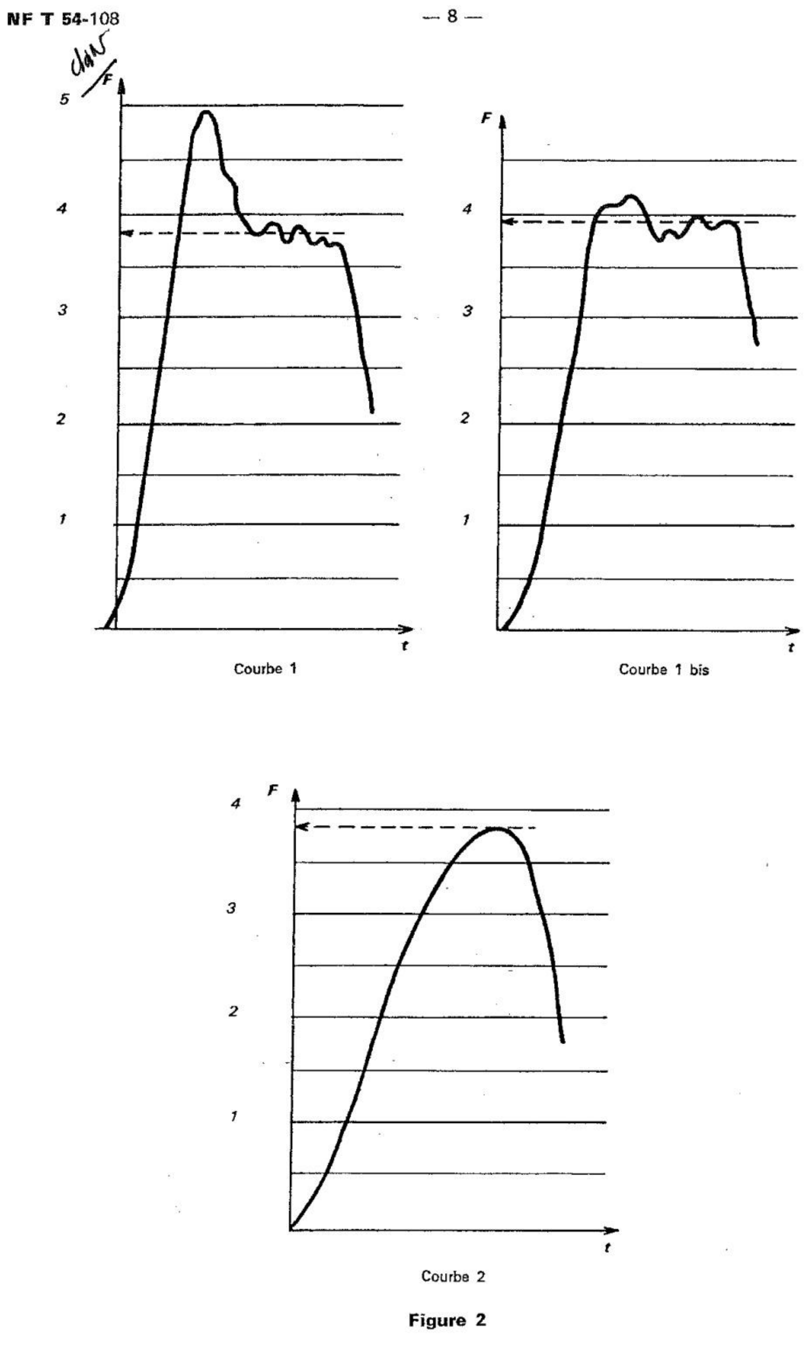

Figure 58 – Typical curves of the behaviour to the resistance against tearing

	Column numbers												
Trial No.	1 A	2 B	3 A×B	4 A×B	5 C	6 A×C	7 A×C	8 B×C	9 D	10 -	11 B×C	12 -	13 -
1	1	1	1	1	1	1	1	1	1	1	1	1	1
2	1	1	1	1	2	2	2	2	2	2	2	2	2
3	1	1	1	1	3	3	3	3	3	3	3	3	3
4	1	2	2	2	1	1	1	2	2	2	3	3	3
5	1	2	2	2	2	2	2	3	3	3	1	1	1
6	1	2	2	2	3	3	3	1	1	1	2	2	2
7	1	3	3	3	1	1	1	3	3	3	2	2	2
8	1	3	3	3	2	2	2	1	1	1	3	3	3
9	1	3	3	3	3	3	3	2	2	2	1	1	1
10	2	1	2	3	1	2	3	1	2	3	1	2	3
11	2	1	2	3	2	3	1	2	3	1	2	3	1
12	2	1	2	3	3	1	2	3	1	2	3	1	2
13	2	2	3	1	1	2	3	2	3	1	3	1	2
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15	2	2	3	1	3	1	2	1	2	3	2	3	1
16	2	3	1	2	1	2	3	3	1	2	2	3	1
17	2	3	1	2	2	3	1	1	2	3	3	1	2
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24	3	2	1	3	3	2	1	1	3	2	2	1	3
25	3	3	2	1	1	3	2	3	2	1	2	1	3
26	3	3	2	1	2	1	3	1	3	2	3	2	1
27	3	3	2	1	3	2	1	2	1	3	1	3	2

Figure 59 – Orthogonal Matrix

1	2	3	4	5	6	7	8	9	10	11	12	13
(1)	3	2	2	6	5	5	9	8	8	12	11	11
	4	4	3	7	7	6	10	10	9	13	13	12
	(2)	1	1	8	9	10	5	6	7	5	6	7
		4	3	11	12	13	11	12	13	8	9	10
		(3)	1	9	10	8	7	5	6	6	7	5
			2	13	11	11	12	13	11	10	8	9
			(4)	10	8	9	6	7	5	7	5	6
				12	13	11	13	11	12	9	10	8
				(5)	1	1	2	3	4	2	4	3
					7	6	11	13	12	8	10	9
					(6)	1	4	2	3	3	2	4
						5	13	12	11	10	9	8
						(7)	3	4	2	4	3	2
							12	11	13	9	8	10
							(8)	1	1	2	3	4
								10	9	5	7	6
								(9)	1	4	2	3
									8	7	6	5
									(10)	3	4	2
										6	5	7
										(11)	1	1
											13	12
											(12)	1
												11

Figure 60 – Triangular table for three level orthogonal arrays (L_{27})