escola superior de tecnologia e gestão
instituto politécnico de leiria

Dissertation Report

Master in Product Design Engineering

Product Design with Scrap Tires

Bilal Chabane Chaouch

Leiria, *September of 2017*

This page was intentionally left blank

Dissertation Report

Master in Product Design Engineering

Product Design with Scrap Tires

Bilal Chabane Chaouch

Dissertation developed under the supervision of Doctor João Matias, professor at the School of Technology and Management of the Polytechnic Institute of Leiria.

Leiria, *September of 2017*

This page was intentionally left blank

Acknowledgements

Vɣiɣ aḍḥemḍaɣ rebi ḍamezwaro, aḍcakraɣ imalaniw ilkoli

syessen ilḥakaɣ assayi ɣwayen iyevɣiɣ

I am willing to express my deep gratitude for; Doctor João Matias, for his guidance, support and supervising during the whole project. For all professor from Product Design Engineering master especially; Doctor Fábio Simões and Doctor Carlos Capela for their constant assistance since the beginning of the master program.

My special thanks for all my friends who always stand-up for me, helped and supported me at all the stages. I am eager to show my gratefulness for Tiger Prod and all the community of Salahifs.

This page was intentionally left blank

Resumo

No presente estudo, Borracha Granulada de Pneu (GTR) feita a partir de resíduos) de pneus foi misturada com Polipropileno (PP) através do processo de injeção, sem recurso a outros aditivos. Foi investigado o efeito do tamanho da partícula do GTR assim como a composição da mistura utilizada. Para isso foram utilizados dois tamanhos de partícula, 0.25mm e 1mm, em três percentagens de diferentes de composição do material.

As amostras foram testadas e analisadas quanto à dureza *Shore*, à impermeabilidade, e às propriedades mecânicas e morfológicas. Os testes realizados indicaram que quanto maior for a dimensão da partícula maiores serão os espaços entre os dois componentes, o que provavelmente é a causa do baixo desempenho verificado quando testados à tração.

A adição de GTR ao PP claramente melhorou a impermeabilidade da mistura resultante de PP/GTR, dando indicações de que pode ser utilizada em trabalhos futuros aplicando conceitos de Design, nomeadamente design para o ambiente, para a reciclagem, para a produção e para o custo.

Palavras-chave: Reciclagem, sucata de pneu, granulado de borracha de pneu (GTR), elastómeros termoplásticos

This page was intentionally left blank

Abstract

In this study; Ground Tire Rubber (GTR) from scrap tires were blended with Polypropylene (PP) using injection moulding without any additional additives.

The effect of particle size and content was investigated by using 0.25mm and 1mm particles with 3 different level contents.

Testing and analysing the samples for shore hardness, wettability, mechanical and morphological properties. The former showed, the bigger the particle size the bigger are the gaps between the phases which is probably the cause for poor tensile performance reported.

Adding GTR to PP clearly enhanced the wettability property, the resultant mixture PP/GTR could be used in future works for achieving Design for Environment, Recycling, Manufacturing and Cost.

Keywords: recycling, scrap tire, Ground Tire Rubber, Thermoplastic elastomers

This page was intentionally left blank

List of figures

Figure 2- 1 Cross section of tire [18] 4

Figure 2- 2 Breakdown of tire ingredients [21] 6

Figure 2- 3 World’s biggest tyre graveyard localised in Kuwait with over 7 million tires [9] 7

Figure 2- 4 TIRE INFERNO; A towering cloud of smoke rises from the Rhinehart tire fire in rural Virginia on Oct. 31, 1983 [12] 8

Figure 2- 5 Flow of waste tire utilisation [13] 9

Figure 2- 6 Hierarchy of waste tire utilization [13] 10

Figure 2- 7 ELT management schemes in Europe [44] 11

Figure 2- 8 Boat bumper and sound absorbing wall [17] 13

Figure 4- 1 Sieving Diagram [78] 19

Figure 4- 2 The sieves column mounted on the shaker base 20

Figure 4- 3 GTR(DUST) sieving analysis test 21

Figure 4- 4 GTR(MIXED) sieving analysis test 21

Figure 4- 5 Werner & Pfleiderer ZSK25x38D extruder 22

Figure 4- 6Granulator and Werner & Pfleiderer ZSK25x38D extruder 23

Figure 4- 7 Boy 22A injection machine 24

Figure 4- 8 Mould used for tensile samplesz 25

Figure 5- 1 Diagram of a durometer indenter or presser foot used for Shores A and D [82] 26

Figure 5- 2 Thermoplastic hardness range[65] 27

Figure 5- 3 Shore D test equipment 28

Figure 5- 4 Illustration of contact angles formed by sessile liquid drops on a smooth homogeneous [36] 29

Figure 5- 5 ATTENSION THETA used for contact angle test 30

Figure 5- 6 (a) Load-elongation curve from a tensile test and (b) corresponding engineering stress-strain curve. Specimen diameter, 12.5 [68] 31

Figure 5- 7 Engineering stress-strain curve showing elastic and plastic deformation [88] 32

Figure 5- 8 Test specimen types 1A and 1B [72] 34

Figure 5- 9 Universal Instron model 4505 used for tensile test 35

Figure 5- 10 SkyScan-Bruker used for micro CT test [78]...................................... 36
Figure 5- 11 Mean of hardness values from different mixtures 37
Figure 5- 12 Shore A-D hardness scale cross references [35].................................. 38
Figure 5- 13 Contact angle mean for different mixtures 39
Figure 5- 14 Tensile strength of different mixtures.. 41
Figure 5- 15 Change in tensile strength values (MPa) in function of GTR content. 42
Figure 5- 16 Elastic modulus of different mixtures.. 42
Figure 5- 17 Elongation at break (in %) of different mixtures.................................. 43
Figure 5- 18 Elongatio at break (%) in function of GTR content.............................. 44
Figure 5- 19 Modulus of toughness of different mixtures... 45
Figure 5- 20 stress vs strain for PP100 (pure PP).. 46
Figure 5- 21 Stress vs Strain for PP70GTR(1mm)30.. 46
Figure 5- 22 Stress vs strain PP50GTR(1mm)50 46
Figure 5- 23 Stress vs Strain PP70GTR(0.25mm)30... 46
Figure 5- 24 Stress vs Strain PP50GTR(0.25mm)50... 46
Figure 5- 25 Stress vs Strain PP30GTR(0.25mm)70... 46
Figure 5- 26 Engineerng Stress-curves of PP/GTR with different content and particle sizes 47
Figure 5- 28 micro-CT result for PP100.. 48
Figure 5- 29 micro-CT result for PP70 GTR(0.25mm) 30.. 49
Figure 5- 30 ImageJ result for PP70 GT(0.25mm)30.. 49
Figure 5- 31 micro-CT result for PP50 GTR(0.25mm)50... 50
Figure 5- 32 ImageJ result for PP50 GTR(0.25mm)50 50
Figure 5- 33 micro-CT result for PP30 GTR(0.25mm)70... 51
Figure 5- 34 ImageJ result for PP30 GTR(0.25mm)70 51
Figure 5- 35 micro-CT result for PP70 GTR(1mm)30.. 52
Figure 5- 36 ImageJ result for PP70GTR(1mm)30 52
Figure 5- 37 micro-CT result for PP50 GTR(1mm)50.. 53
Figure 5- 38 ImageJ result for PP50 GTR(1mm)50 53
Figure 7- 1 Robot arm used for Additive Manufacturing.. 55
Figure 7- 2 3D model used for Additive Manufacturing... 55
Figure 7- 3 Final result of Additive Manufacturing 56

This page was intentionally left blank

List of tables

Table 2- 1 Typical materials used in tyre manufacturing (in Europe and USA) according to the percentage of the total weight of the finished tyre that each material class represents [14] 9
Table 2- 2 General methods of waste tire downsizing [3] 13
Table 4- 1 Mixtures prepared for processing 22
Table 4- 2 Extrusion temperatures in each zone 23
Table 4- 3 Injection machine parameters [75] 24
Table 4- 4 Injection parameters used for each mixture 25
Table 5- 1 Shore Test conditions 28
Table 5- 2 Shore D hardness values of different mixtures 37
Table 5- 3 Contact angles values for different mixtures 39
Table 5- 4 Tensile test result for different mixtures 41

List of acronyms

ETRMA	ETRMA-European Tyre & Rubber Manufacturers' Association
RMA	Rubber Manufacturers Association
JATMA	Japan Automobile Tyre Manufacturers Association
WVSCD	World Business Council for Sustainable Development
GTR	Ground Tire Rubber
PP	Polypropylene
ELT	End of Life Rubber
WTR	Waste Tire Rubber
TPE	Thermoplastic elastomer
RTR	Reclaimed Tire Rubber
DGTR	Devulcanized Ground Tire Rubber
CDRSP	Centro para o Desenvolvimento Rápido e Sustentavel do Produto
IPL	Instituto Politécnico de Leiria
IP	Injection Pressure
IV	Injection Velocity
2IP	2nd Injection Pressure
HT	Holding Time
IT	Injection Time
wt%	Weight percent
ASTM	American Society for Testing and Materials
ISO	International Organization for Standardization
γ_{lv}	Liquid-vapor interfacial tension
γ_{sv}	Solid-vapor interfacial tension
γ_{sl}	Solid-liquid interfacial tension
θ_Y	Contact (or Young's) angle
s	Engineering (or nominal) stress
F	Tensile force
A0	Initial cross-sectional area of the gauge
e	Engineering (or nominal) strain
ΔL	Change in gauge length
L0	Initial gauge length

E	Elastic modulus
s’	True stress
A	Actual (instant) area
e’	True strain
Ut	Modulus of toughness
CT	Computed Tomography
STD	Standard deviation
WPE	Waste Polyethylene
EB	Elongation at break
LDPE	Low-Density Polyethylene
EPDM	Ethylene-Propylene Diene Monomer
GD&T	Geometric Dimensioning and Tolerancing

This page was intentionally left blank

Table of Contents

ACKNOWLEDGEMENTS III

RESUMO V

ABSTRACT VII

LIST OF FIGURES IX

LIST OF TABLES XII

LIST OF ACRONYMS XIII

TABLE OF CONTENTS XVI

1. INTRODUCTION 1

2. RESEARCH BACKGROUND 4

2.1. Tire Composition 4

2.2. Discarded Tire Problematic 6

2.3. Waste Tire Reuse 8
  2.3.1. Management of Used Tires in Europe 10
  2.3.2. Recycling Waste Tires 12

2.4. Waste Tire Applications 13

3. GTR AND RTR IN POLYMER BLENDS 16

3.1. GTR in Thermoplastics 16

3.2. GTR in thermosets 17

3.3. GTR in rubbers 17

3.4. Plastic and rubber compatibility 17

4. TEST SAMPLES' PREPARATION 19

4.1. Materials Preparation 19

4.2. Materials processing 21

4.2.1. Mixtures preparation 21
4.2.2. Extrusion process 22
4.2.3. Injection moulding 24

5. CHARACTERIZATION TECHNIQUES AND TESTS 26

5.1. Hardness Test 26
5.1.1. Shore (Durometer) Hardness 26
5.1.2. Test Procedure (ISO 868) 27

5.2. Contact Angle (wettability) Test 29

5.3. Tensile test (ISO 527) 30
5.3.1. Tensile specimens 33
5.3.2. Tensile machine 34

5.4. Morphology Test 35

6. RESULTS AND DISCUSSION 37

6.1. Shore Hardness Test 37

6.2. Contact Angle Test 39

6.3. Tensile Test 40

6.3. Morphology Test 48

7. CONCLUSIONS 54

REFERENCES 58

APPENDICES 68

Appendix A 68

Appendix B 69

Appendix C 70

1. Introduction

The energy crisis and environmental degradation are amongst the main problems mankind is facing today. These problems owe their origin to a growing population, rapid industrialization and huge quantities of solid refuse, which are generated daily. It has become imperative to make use of appropriate technologies to alleviate part of our energy crisis and environmental degradation for the possible recovery of resources from non-conventional sources, like municipal and/or industrial wastes, refused plastics, scrap tires and others. The disposal of these solid wastes from human activity is a growing environmental problem for modern society, especially in developing countries [1].

Back to history, there have always been elements of re-use, and some industries or sectors have always reused raw materials. In fact, until the industrial revolution "re-use until the point of no return" was probably the norm. Wood, stone, metal, clothing etc., were all reused until they had no value or use left. Then the time of the industrial revolution arrived and it was discovered how to increase production rate and efficiency, they could make many things quickly and cheaply and the society became an increasingly throwaway society. Until a day where landfills became open mines for resources such as rubber, plastics and other long lived materials [2], [3].

Although scrap tires account for only 2wt% of total waste [4], their management is attracting growing interest in developed countries. This interest might be the consequence of the environmental problems they may generate through inappropriate management, on one hand, on the other with the increasing demand for recycling raw materials. European Union has introduced directives for the management of scrap tires including the European commission’s landfill Directive (1999). The commission has banned the disposal of scrap tires to landfill since 2006 and the European End Life Vehicle Directive 2000 which requires that the 80% in weight of an end of life vehicle must be recycled [5],[6],[7]. In another meaning, it proposes the following routine: to reduce, to reuse, to recycle, and finally, to perform an energy valorisation process. Although it would be desirable to implement only the three first strategies, waste tire generation is so high that it is unavoidable to make use of energy valorisation process. In Europe, the main methods for scrap tires management are materials recovery (38.7%), energy recovery (32.3%) or retreading (11.3%) [8]. But globally, wide spread in the world, approximately 64% of used tires went to landfill or were illegally dumped or stockpiled, while only 13% were recycled [8],[9].

According to the analysis of Sienkiewicz et al. [10] on the analysis done to the reports issued by the largest associations of tyres (ETRMA [2], RMA [11], JATMA [12],WBCSD [13]), the growth in the number of used tires around the globe is becoming alarming and rapid measures should be taken.

At each stage of product design, decisions about the materials from which the product will be made is required and as well the production process corresponding to that material. Final product cost is directly influenced by both materials and the process. The engineer, to make his decisions needs the characteristics of that material and the process corresponding [14]. To develop products with scrap tires, hence, it is required first to study the material and the process.

Based on this latter and the scrap tires environment effect, the present study took its motivations on the sake of synthesizing cheap & eco-friendly thermoplastic based on ground tire rubber (GTR). It was chosen Polypropylene (PP) as the polymer matrix following its proven properties, processability and price. An injection moulding as the process, since Scaffaro *et al.* [15] had proven that the samples produced with injection machining (because of additional mixing in the fuse and the compacting force) offers better properties than compression moulding.

This report highlights how thermoplastic based on GTR can be considered for manufacturing products while complying with principals of Design for Environment and for Recycling (since it is based on reusing waste in spite of a raw material) and Design for Manufacturing and for Cost (since it is based on injection moulding “of waste-cheap material” which by itself can reach massive production rate at small cost).

The main objective of the study is, while injecting PP/GTR of two different particle sizes using different percentages of GTR, to investigate the effect of PP/GTR rubber particle size and composition into, shore hardness, wettability, mechanical and morphological properties.

This report is divided into seven main chapters as following explained:

1- Introduction; addressing waste tire issue in general and how the study is aiming to contribute to the solution.

2- Research Background; a literature review is presented about tire composition, the problematic of wasted tires, the present use of wasted tire, recycling methods and its management in Europe.

3- GTR & RTR in Polymer Blends; current of past work on blending tire rubber with different polymers (thermoplastics, thermosets and rubbers).

4- Test Samples' Preparation: summarizes the materials and the processing methods used.

5- Characterization Techniques and Tests; explains different test performed and their respective processed methods.

6- Results and Discussion: shows the results of the tests performed while comparing with relevant works.

7- Conclusion; summarizes the conclusions on the results and addresses the tasks that could be performed in future.

2. Research Background

2.1. Tire Composition

In a technical definition; a tire is a hollow-round shape covering the perimeter of a wheel. It is on most ground vehicles to protect the wheel from wear and tear as well as to provide the friction between the vehicle and the road needed for acceleration and handling. The main functions of tire are supporting vehicle loading, transmits vehicle propulsion and braking, maintaining or changing the vehicle direction, and absorbing road shocks [16], [17]. A tire skeleton is composed of several parts as can be seen form Figure 2- 1 which depicts a tire cross section.

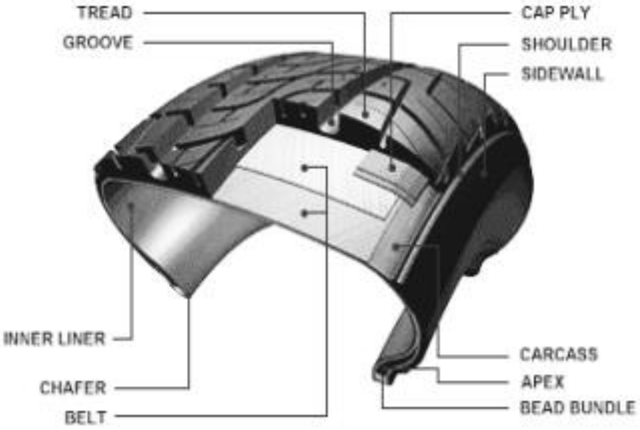


Figure 2- 1 Cross section of tire [18]

The tire parts as shown in Figure 2- 1 are [17]:

- **Tread:** is the part which is in direct contact with the road surface. It is made of rubber with additions of carbon black and other chemicals.
- **Groove:** It is kind of a channel within the tread. A tire groove improves driving stability, pulling and braking ability and cooling down the rubber.
- **Cap ply:** are steel cords stabilize the structure of the tire against internal pressure.
- **Shoulder:** is the section of transition between the edge of the tread and the sidewall.
- **Sidewall:** can be considered as a bridge between tread and bead, often strengthened with steel cords or fabric to provide strength and flexibility.
- **Carcass:** is the tire frame which provides coherence, made of fabric embedded in rubber can be added steel cords.

- **Apex:** is filler providing buffer between bead, inner liner and body ply.
- **Bead bundle:** are rigid bands of steel wire in rubber compound, it contributes to press the tire to fit on the wheel.
- **Belt:** are layers of rubber and meticulously spaced steel cords, provide strength while maintaining the flexibility.
- **Chafer:** is a protection of the steel cords against wear and cutting by the rim, as well as it prevents dirt and moisture from getting into the rim.
- **Inner liner:** is an air-tight rubber sheet made to hold high-pressure air inside.

Tires are made of vulcanized rubber and various reinforcing materials. Natural and synthetic rubbers are commonly used in tires for different purposes. Natural rubber is an essential element of a tyre since it has unique elastic properties. Different types of synthetic rubbers are added to natural rubber to achieve the desired elasticity [17]. Figure 2- 2 depicts the ingredients of a tire which are, in addition to the rubber compound [19], [20]:

- Reinforcing fillers such as carbon black. It is used to strengthen the rubber and make tire have more abrasion resistance and also UV protection from sun light;
- Reinforcing fibres such as textile or steel fibres. It is usually in the form of a cord which is used to provide the reinforcing strength in tires;
- Extenders such as petroleum oils; are used to control the viscosity, reduce internal friction during processing, and improve low temperature flexibility in the vulcanized product;
- Vulcanizing agents such as organ-sulphur compounds. It is used as the chemical agent for the vulcanization process;
- Zinc oxide and stearic acid, used to activate the curing (cross-linking) system and to preserve cured properties.

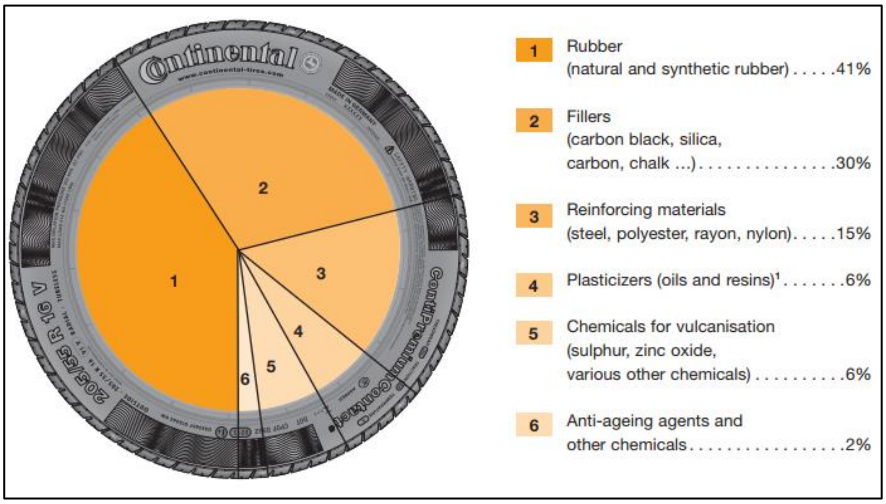


Figure 2- 2 Breakdown of tire ingredients [21]

2.2. Discarded Tire Problematic

In the report of ETRMA of 2015 [2], it was considered that scrap tire recycling (or END-OF-LIFE tire recycling) is a global issue, and how it will be used is both a challenge and an opportunity.

The challenge is derived from many directions, as it can be seen from recent developments it is not because of a lack of ideas, rather than the implementation of those ideas. The great opportunity is the use of these tires recycled rubber that can fit in many sectors such as; construction, urban furniture, traffic accessories and others.

Sienkiewicz *et al.* [10] reported the yearly amount of used tires is about 17 million tonnes matching the annual production of 1.4 billion. The amount becomes a serious problem when landfilling is being used for tire disposal.

Landfill, which is a site where a disposed waste from different sources are buried [22]. Landfilling is considered as the earliest way on how-to-deal with scrap tires, nonetheless still is one of the most undesirable methods, as it causes severe environmental problems [23], [24].

The shape and impermeability property of discarded tires allow it to hold water for a long period of time. Thus, providing sites for mosquito larva breeding, which can provoke deadly diseases such as dengue, chikungunya and malaria. Disposed tire can also provide breeding ground to other animals such as snakes [25], [26]. Figure 2- 3 shows an aerial view of what is considered as the world's biggest tyre graveyard localised in the desert of Kuwait, where is disposed over 7 million scrap tires [27].

Figure 2- 3 World's biggest tyre graveyard localised in Kuwait with over 7 million tires [9]

Moreover, disposed tire increases fire threat especially during hot periods. According to Jang et al. [28], a whole disposed tire represents an empty space of 75% which makes it difficult to either quench the fire with water or cut off oxygen supply. Because of the composition of tires, tire-fire can cause surface, subsurface, water and soils contaminations. The fire smoke includes dense black smoke which pollutes the air with toxic gas emissions and impairs visibility [29].

The example of this dangerous situation was the ***Tire Inferno*** fire. It took place at storage facility in Rhinehart, Northern Virginia USA, it started on October, 31th, 1983, where it was disposed nearly 7 million tires. Tire inferno, also called the Rhinehart tire fire, burned for nine months! Nearby water sources were polluted with lead and arsenic compounds and it took about 22 years and 12 million US dollars to clean [30][31]. Figure 2- 4 shows the smoke released by the "Tire Inferno".

Figure 2- 4 TIRE INFERNO; A towering cloud of smoke rises from the Rhinehart tire fire in rural Virginia on Oct. 31, 1983 [12]

Day *et al.* [32] stated that; eco-toxicity is likewise affected in consequence of discarded tires release of metals and additives such as stabilizers, flame retardants, colourants and plasticizers. Leaching of these materials into the soil and the water is definitely not eco-friendly as it could possibly kill the bio-organic colonies [33].

2.3. Waste Tire Reuse

As cited by Ramarad et al. [34], recovered waste tires are considered to be rich materials because of their compositions and properties, hence waste tire is a valuable source of raw materials as can be seen on Table 2- 1.

Table 2- 1 Typical materials used in tyre manufacturing (in Europe and USA) according to the percentage of the total weight of the finished tyre that each material class represents [14]

Materials	In USA		In European Union	
	Passenger tyre	Truck tyre	Passenger tyre	Truck tyre
Natural rubber (%)	14	27	22	30
Synthetic rubber (%)	27	14	23	15
Carbon black (%)	28	28	28	20
Steel (%)	14–15	14–15	13	25
Fabric, fillers, accelerators, antiozonants, etc. (%)	16–17	16–17	14	10
Average weight	New 11 kg, Scrap 9 kg	New 54 kg, Scrap 45 kg	New 8,5 kg, Scrap 7 kg	New 65 kg, Scrap 56 kg

Figure 2- 5 illustrates the flow of waste tire utilisation.

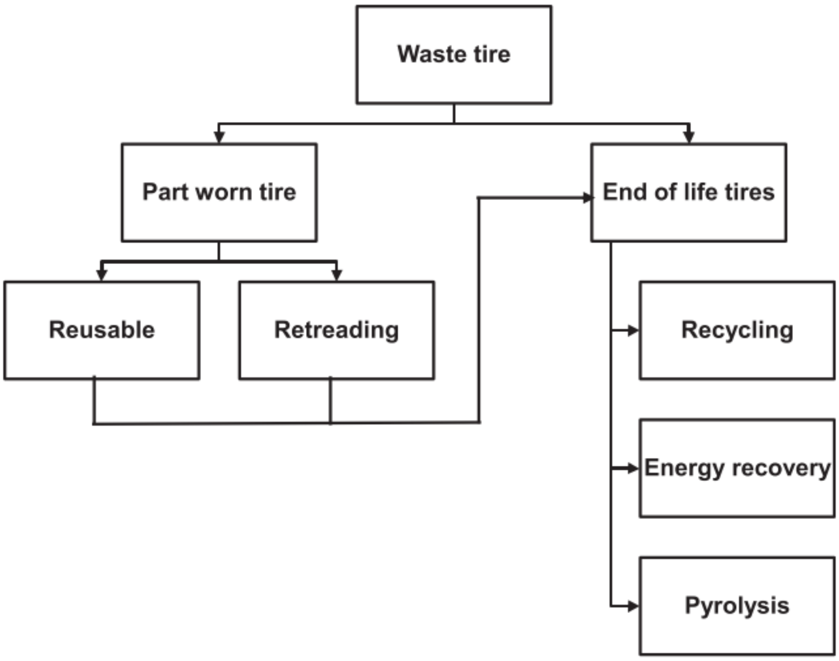


Figure 2- 5 Flow of waste tire utilisation [13]

These waste tires can be separated into part worn tires and end of life tires (ELTs). Part worn tires are still suitable for road use, while end-of-life-tires are not anymore. Numerous attempts have been made to utilize ELTs by reclamation, devulcanization, energy recovery, pyrolysis and others [19], [25], [35]–[38].

As suggested by Ramarad *et al.* [39], the ideal waste tire utilisation hierarchy is as depicted in Figure 2- 6;

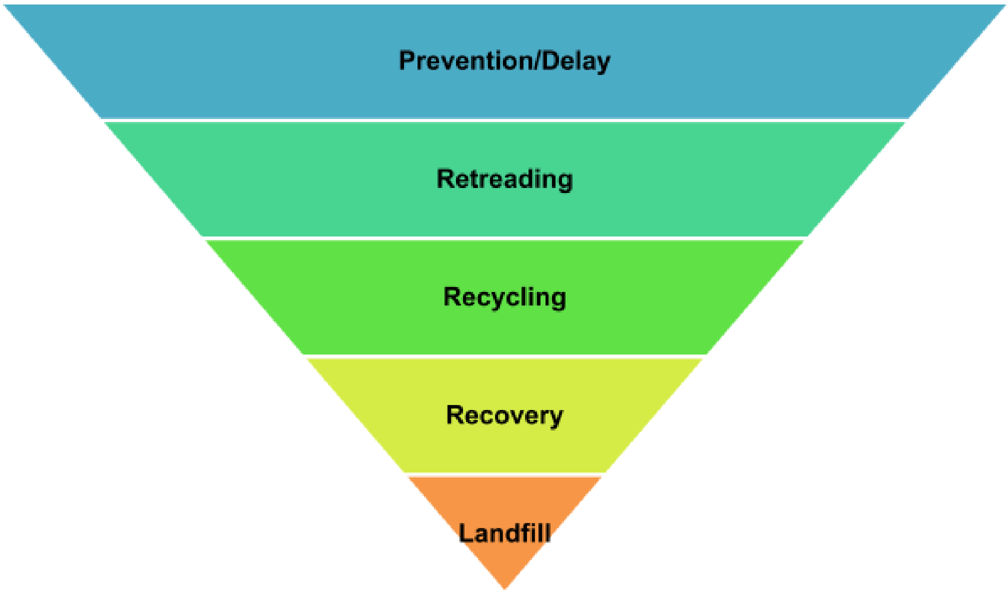


Figure 2- 6 Hierarchy of waste tire utilization [13]

First and foremost, waste tire generation should be prevented. In an article, van Beukering *et al.* [40] cited that correctly inflated, rotated and cared tires would last 50 to 90% longer on the road.

Then is retreading, which is kind of re-manufacturing method by replacing the tread on worn tires [41]. When a retreaded tire enters the market, it reduces the manufacturing of the new one. Ferrer in his article "The economics of tire remanufacturing" [42], declared that retreading is the most economically worthwhile method of waste tire utilization as it requires only 30% of energy and 25% of raw materials used in the manufacturing of a new tire.

In third position comes recycling, followed by recovery. Currently, recycling and recovery are the most used methods [34]. Recycling should be chosen over recovery, as recovery method is only capable of recovering 30 to 38% of energy embedded in new tires [26], [43].

At last, which is the most undesired method (with all the issues associated and described in previous sections), is landfill [34].

2.3.1. Management of Used Tires in Europe

As a result of European Directive 1999/31/EC dated 26 April 1999, it is forbidden by the law to dispose used tires in landfills. The directive instructs state members to focus on responsible and environment-friendly recovery methods [44].

There are no explicit European regulations for recovering used tires. Each state member is free to choose its own management system from the three available types of management systems which are illustrated in Figure 2- 7 [44] :

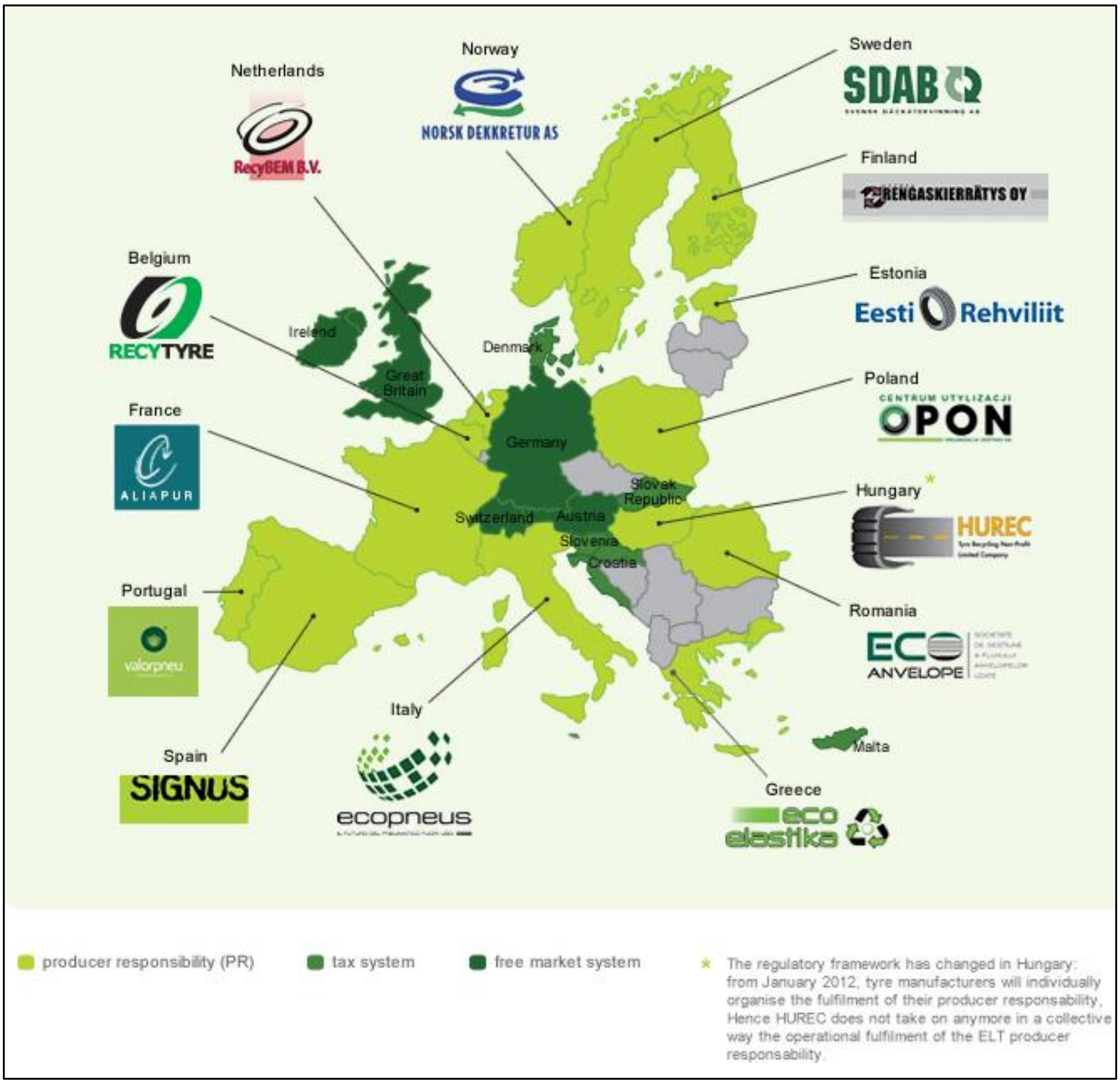


Figure 2- 7 ELT management schemes in Europe [44]

a) **The producer responsibility system:** used particularly in Portugal, under that system, the law defines the legal framework and assigns the duty of ELTs management to the tire producer (manufacturer or importer). This makes the producer responsible for the waste that the consumer generates. [2]. In Portugal, this contribution can be found in the invoice of a new tire [44].

b) **The tax system:** Under this system, each country is responsible for the management of ELTs. The process is financed by a tax collected from tire producers and subsequently from the consumer [2].

c) **The free market system:** under this system, also referred as liberal system, the legislation defines the objectives to be met but at the same time does not designate the responsible. In this way, all the operators are implicated in the recovery chain and act in obedience with the legislation [2].

2.3.2. Recycling Waste Tires

For this work, the chosen waste tire utilisation method would be recycling as it is a valuable source of raw material.

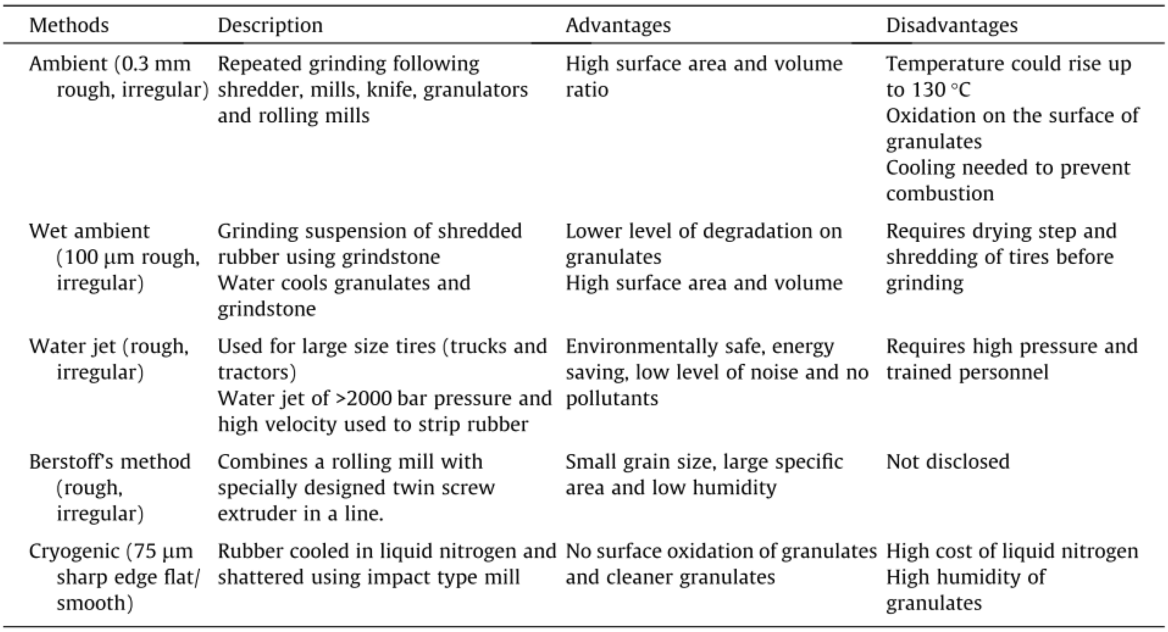
Products that are usually manufactured with a “conventional” materials as wood and metal, are nowadays being produced with polymeric. Waste tire, containing almost 50% rubber is considered a polymeric material commonly called waste tire rubber (WTR) [34].

Blending WTR with other polymers also backs the 3R notion (reduce, reuse and recycle). Using WTR, it would result on reducing the amount of virgin polymer used and recycling the waste tires already exist. These factors have motivated many scientists and researchers around the world to create and evaluate this wasted material [45].

Downsizing waste tires: Downsizing waste tires is done by chopping the tire into smaller sized particles [46], this process is mandatory for blending waste tire rubber with polymers [19]. Sunthonpagasit and Duffey [47] considered that downsizing waste tires requires special machinery and equipment capable of grinding waste tires with complex structure and high mechanical properties (which reported in the previous sections).

Table 2- 2 illustrates the main methods of waste tire downsizing with their advantages and disadvantages. With the granulation process, the textiles and steels from the tires will be removed by pneumatic separators and electromagnets respectively [47]. This process, downsizing waste tires, will result on ground tire rubber (GTR).

Table 2- 2 General methods of waste tire downsizing [3]



Methods	Description	Advantages	Disadvantages
Ambient (0.3 mm rough, irregular)	Repeated grinding following shredder, mills, knife, granulators and rolling mills	High surface area and volume ratio	Temperature could rise up to 130 °C Oxidation on the surface of granulates Cooling needed to prevent combustion
Wet ambient (100 μm rough, irregular)	Grinding suspension of shredded rubber using grindstone Water cools granulates and grindstone	Lower level of degradation on granulates High surface area and volume	Requires drying step and shredding of tires before grinding
Water jet (rough, irregular)	Used for large size tires (trucks and tractors) Water jet of >2000 bar pressure and high velocity used to strip rubber	Environmentally safe, energy saving, low level of noise and no pollutants	Requires high pressure and trained personnel
Berstoff's method (rough, irregular)	Combines a rolling mill with specially designed twin screw extruder in a line.	Small grain size, large specific area and low humidity	Not disclosed
Cryogenic (75 μm sharp edge flat/ smooth)	Rubber cooled in liquid nitrogen and shattered using impact type mill	No surface oxidation of granulates and cleaner granulates	High cost of liquid nitrogen High humidity of granulates

2.4. Waste Tire Applications

The waste tire can be used for environmentally safe application in terms of material and energy recovery instead of being disposed in the environment.

Figure 2- 8 Boat bumper and sound absorbing wall [17]

According to Tasawan [17] and Northeast Recycling Council [48], the most important use of recycled tire material can be summarized into the following applications;

- **Whole tire:** Tires are excellent materials for the original shape because of their resilience to impact and longevity. The whole tire can be used for civil engineering projects such as highway crash barriers, sound absorbing walls, boat bumper, dyke protections, insulation in building foundations and road base material [49]. Two examples of whole tire applications are shown in Figure 2- 8.
- **Gravel Substitute:** wherever gravel is used, chances are that tire chips can be used instead. Applications include: sub-layers for roadways, aggregate for drainage

ditches, and highway embankment backfill and are being used under light rail tracks in a urban area, to reduce vibration and noise from passing trains [50].

- **Crumb Rubber:** Crumb rubber is finely ground rubber produced from waste tires. Down-cycling of tire occurs as discussed in previous section. Some applications for crumb rubber include: rubberized asphalt, playground flooring, welcome mats, anti-fatigue mats and vehicle mud guards [20], [51].
- **Landfill Medium:** Shredded or chipped tires can be used as both a liner or cover for landfills. Tire chips can be used as a thermal insulation between primary and secondary landfill liners to reduce temperatures [52].
- **Wastewater Treatment Filters:** Another use is filters in wastewater treatment and constructed wetlands. Because tires can be chipped to be more porous than organic compounds, rocks and other material, they often serve as a much better filter media [53], [54].
- **Garden Mulch:** Yet another innovative use for recycled tires is rubber mulch. Not only does it hold its appearance over its life cycle, it doesn’t float away during rainstorms, nor will it rot the way wood mulch will. Unlike wood mulch, tire chips don’t breed termites and other wood-boring pests [55].

Other applications are being studied and developed as; reprocessing the tire rubber by injection or compression moulding, with or without the addition of virgin rubber. Guglielmotti *et al* [56] proposed producing rubber parts by tire recycling without using virgin materials. Lima *et al* [57]–[59] studied different properties of GTR in TPE blends for injection moulding purposes and concluded that; this process is a proven practical method to recycle waste tires or what they called “*a potentially wasted material*”.

Modification of GTR: Incorporation of GTR into fresh/virgin polymer can be considered as a tempting-feasible process. GTR contains high quality natural and synthetic rubber which could be used in other polymers such as rubbers, thermoplastic and thermosets. Nonetheless, the compatibility of GTR and polymeric matrix is a weighty matter [34], [45]. Because lack of bonding between GTR and the virgin matrix, those blends usually have lower mechanical properties [46].

On this matter, to improve those properties, the surface of GTR can be modified. Many processes have been developed to address this issue namely;

- **Physical and chemical methods**: there are many methods such as; graft modification, radiation induced modification, gas modification among others [60], [61].
- **Reclaiming or devulcanization:** even if they are different processes the aim is shared, which is obtaining a rubber compound similar to a virgin/fresh one which will be called in this case reclaimed tire rubber (RTR) [25], [43].

3. GTR and RTR in Polymer Blends

The rubber (GTR-Ground Tire Rubber or RTR-Retreated Tire Rubber) extracted from tires, can be incorporated into the three groups of polymers; thermoset, thermoplastic and rubber [62], [63].

Since the polymers market is immensely big and continuously growing, incorporating as little as can be of GTR in polymers, particularly in thermoplastic, would result a big consumption of waste tires [64], [65]. The use of GTR in each polymer category has targeted a precise goal, for example; thermosets industry is interested in improving toughness, thermoplastic industry wants to attain thermoplastic elastomers while rubber sees for cheap filler [66].

3.1. GTR in Thermoplastics

Synthesizing cheap thermoplastic elastomers (TPE) is the powering reason behind the use of GTR in TPEs [66]. The use of GTR in thermoplastics was the often preferred way of recycling besides rubbers [64]. This was supported by the following patterns [58], [64], [65]:

- As reported by Karger-Kocsis *et al.* [64] and Marvin et al [65]; a small percentage of GTR in thermoplastics (less than 10% in weight) can cause a large consumption of GTR owning to the market share of thermoplastics,
- Recycled thermoplastics can be found in the market hence when GTR is added, the final cost is reduced even further,
- The toughness of the corresponding blend may be enhanced as GTR is incorporated. This expectation is since the majority of toughened thermoplastics are rubber modified.

However, the low elongation at break came as a limiting factor [66]. To overcome this issue some recent investigations merged on composites of polymer / GTR or RTR [67], [68]. Reinforcing fillers such as short fibres and nanoparticles are known to increase polymer system's strength. According to Martins *et al.* [68] in their work on sisal fibres and tire rubber composites, the results of the study are encouraging and indicating that this composite offers a promising potential for non-structural applications.

3.2. GTR in thermosets

It is known that rubber particles when disperd in micron-scale may improve the fracture toughness of the respective matrix, and the incorporation of GTR particles in thermoset resins often targeted their toughness improvement [64]. In a study performed by Aoudia *et al.* [69], incorporating GTR and DGTR (micro wave Devulcanized GTR) into an epoxy matrix as reinforcement particles and the obtained composites were characterized by three-point flexural tests. As a result of microwave devulcanization, epoxy composites filled with DGTR had better mechanical properties than those filled with GTR, at the same loading.

According to Karger-Koscis *et al.* in their review [64], the modification of thermosets with GTR is less promising because the desired toughening cannot be achieved. This statement may change when ultrafine GTR particles (≤ 10 µm) are being used. It is worthy to state that the studies conducted on incorporation of GTR in thermosets did not gave a big importance to other properties which may be of interest, such as shrinkage and change in the residual stress state.

3.3. GTR in rubbers

The incorporation of GTR in rubber mixes in appreciable quantities changes the processability and it is associated with some decrease in the mechanical properties, such as tensile and shear strength [70]. Nonetheless, there are has been always considerable interest to recycle GTR in rubber stocks [71], [72].

A recent work investigated by Guglimotti *et al.* [56], on what they call "*Direct Powder Moulding*", it consists on producing large rubber parts from spent tyres without any addition of virgin materials or linking agents. The rubber pads were produced by compression moulding of rubber powder mixtures which were obtained by mechanical grinding of ground tyre rubbers. In this study, the effect of different powder mixtures on the final performances of the moulded parts was evaluated, and it was found that the rubber powder distribution strongly affects the mechanical performances of the recycled rubber moulded products [73].

3.4. Plastic and rubber compatibility

As referred previously, compounding GTR with thermoplastic polymers is a cost-efficient way to prepare TPEs. The principal issue is on improving the compatibility between the vulcanized rubber particles and plastic matrix to obtain type of isotropic morphology that would result on enhancement of mechanical properties [74].

According to D. Mangaraj [75], the role of the compatibilization is:

1) Reducing the interfacial energy to improve adhesion between phases;
2) Obtaining better dispersion during mixing;
3) Stabilizing dispersion against agglomeration during processing;
4) For GTR-thermoplastic polymer, smaller particle size of GTR and de-agglomeration are the most important factors in manufacturing ideal materials.

According to Liu [74], since GTR particles are crosslinked structures and they cannot be reprocessed unless through devulcanization [75], and adding compatibilizers would not achieve the desired enhancement of properties. Thus compatibilization of GTR-TPE blends can be accomplished by using a reduced size of GTR particles to increase specific surface [74].

The annual consumption of TPEs is growing. So, the development of GTR-containing TPEs with useful characteristics would contribute to the recycling of a large amount of waste tire rubber. Karger-Kocsis *et al* [64] concluded that GTR recycling should target the production of TPEs and its usage in the rubber industry.

4. Test Samples' Preparation

4.1. Materials Preparation

The polymer matrix used in the mixtures was a polypropylene supplied by REPSOL, PP 070 G2M. Initially it was used three different types of particle sizes GTR;

- a powder-like referred at first as DUST,
- a mixed-sizes that seemed to have different particles with different sizes referred at first as MIXED
- and the third is 2.4mm particles size.

The GTR were supplied by different partners of CDRSP-IPLeiria, it was needed to measure the particle size of two samples; DUST and MIXED.

The particle size measuring test, also called Sieve analysis or Gradation test [76], was done in the laboratory of *Geotecnia e Vias de Comunicação* from civil engineering department of ESTG. The test involves a nested column of Sieves with wire mesh cloth (screen) [77].

A previously weighed sample is poured into the top sieve which has the largest screen openings. Each lower sieve in the column has smaller openings than the one above. At the base is a round pan called the receiver, to collect the rest if there was any [77], as can be seen on Figure 4- 1.

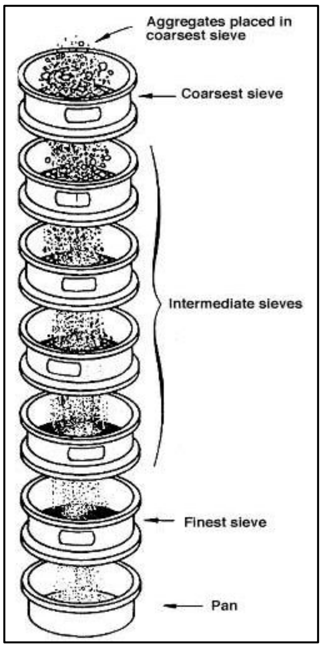


Figure 4- 1 Sieving Diagram [78]

The column is placed on a mechanical shaker. The shaker vibrates the column for 2 minutes period. After the shaking is complete the material on each sieve is weighed. The weight of the sample of each sieve is then divided by the total weight to give the respective percentage retained. The size of the average particle on each sieve is then analysed to get a cut-off point or specific size range. The results are plotted and displayed on Figure 4- 3 and Figure 4- 4.

For GTR-DUST, the most dominant particle size is 0.250mm (250 µm) with 62.21% and there is the presence of 0.125mm and 0.063mm particle with 24.92% and 8.32% respectively.

For GTR-MIXED, the most dominant particle size is 1.000mm with 65.25%, and as well the presence of, beside to smaller size <0.250 which can be considered insignificant, of 2.000mm particle with 14.30% and of 0.5mm (500µm) with 16.25% were observed. Hence from now on, the GTR-DUST will be represented as GTR(0.25mm), and the GTR-MIXED as GTR (1mm).

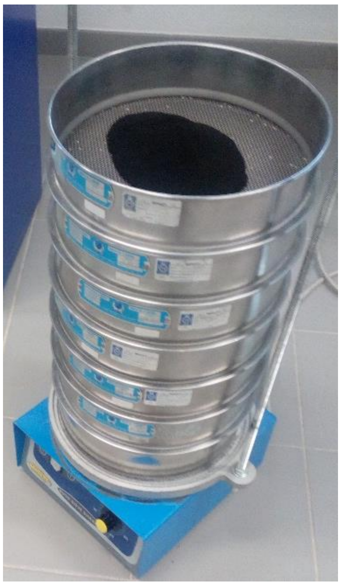


Figure 4- 2 The sieves column mounted on the shaker base

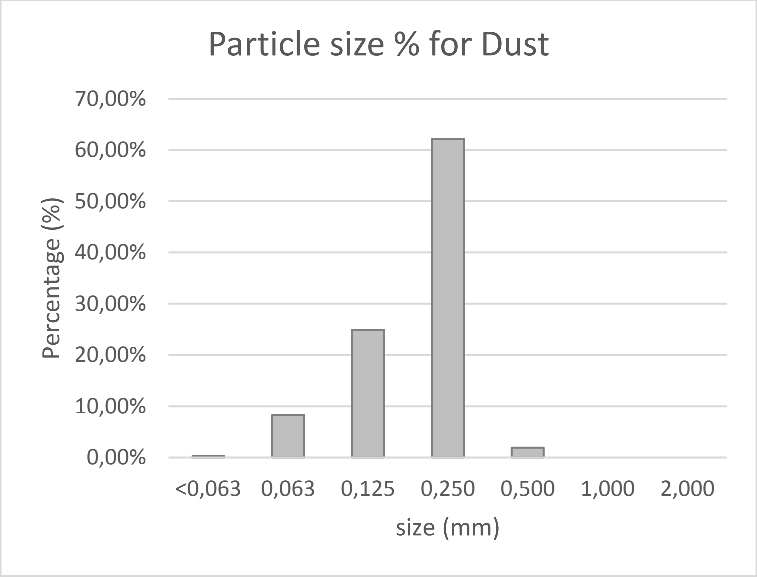


Figure 4- 3 GTR(DUST) sieving analysis test

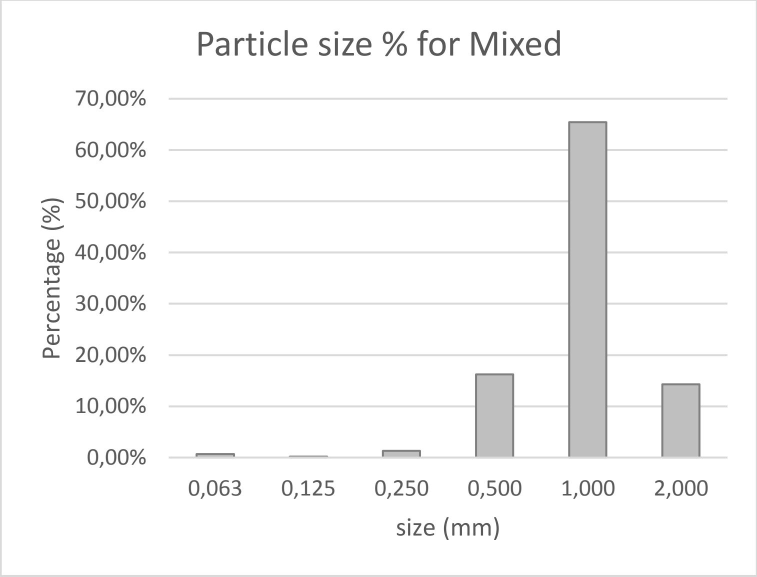


Figure 4- 4 GTR(MIXED) sieving analysis test

4.2. Materials processing

4.2.1. Mixtures preparation

First the mixtures were prepared with three different GTR percentage content (30%, 50% and 70%) for each particle size. It was prepared 1 kg of each PP/GTR mixtures shown in Table 4- 1.

Table 4- 1 Mixtures prepared for processing

MIXTURES	DESIGNATION	PP wt% content	GTR wt% content
1	PP100	100wt%	0wt%
2	PP70 GTR(0.25mm) 30	70wt%	30wt%
3	PP50 GTR(0.25mm) 50	50wt%	50wt%
4	PP30 GTR(0.25mm) 70	30wt%	70wt%
5	PP70 GTR(1mm) 30	70wt%	30wt%
6	PP50 GTR(1mm) 50	50wt%	50wt%
7	PP30 GTR(1mm) 70	30wt%	70wt%
8	PP70 GTR(2.4mm)30	70wt%	30wt%
9	PP50 GTR(2.4mm) 50	50wt%	50wt%
10	PP30 GTR(2.4mm)70	30wt%	70wt%

4.2.2. Extrusion process

The extrusion was done using a Werner & Pfleiderer ZSK25x38D extruder available in laboratory of mechanical technologies of CDRSP-IPL depicted in Figure 4- 5. The extruder is constituted of extruding diameter of 3mm, a motor with a power of 10 kw and a maximum rotation of 300 rpm. The equipment is also equipped of a water tank with rollers to cool down the extruded filament and a granulator (Figure 4- 6) [79], [80].

Figure 4- 5 Werner & Pfleiderer ZSK25x38D extruder

The mixtures were extruded under a specified temperature on each zone as depicted on Table 4- 2. The velocity of the first screw was 40 rpm and for the second screw was 30rpm. On second plan the extruded filament was granulated and packed in labelled-sealed bags.

Table 4- 2 Extrusion temperatures in each zone

Zone 1	Zone 2	Zone 3	Zone 4	Zone 5	Zone 6	Zone 7	Zone 8	Zone 9
140	140	170	170	180	180	180	180	180

Figure 4- 6Granulator and Werner & Pfleiderer ZSK25x38D extruder

At this stage, it was noticed that some mixtures namely; PP30 GTR(1mm) 70, and all the three with 2.4 mm particle size were not processable (the extruded filament keep breaking up to small portions even when changing extrusion parameters, which makes them harder to granulate). Probably, this was due to the 3 mm diameter of the fuse; having particles with 2.4mm leaves small spaces for the PP and this way it is impossible to promote the adhesion in-between materials causing the mixture to be constantly breaking during the extrusion process. By the other hand, with 70wt% of 1mm (which has 14.3% with 2mm) could not be extruded using a 3mm diameter fuse under the conditions used during the process due to the high viscosity of the mixture.

After the extrusion, all the mixtures were dried at 60°C for 8 hours.

4.2.3. Injection moulding

a) **Injection machine:** To produce the tensile samples, a micro-injection machine was used in Figure 4- 7, Boy 22A, available in the same laboratory as the extruder. The machine is designed for the automatic injection of thermoplastics, elastomers, silicones as well as other polymers with a closing force of 1000 kN [79], [81].

Figure 4- 7 Boy 22A injection machine

The Boy-22A injection machine has the following characteristics;

Table 4- 3 Injection machine parameters [79]

Injection Side		Ejection Side	
Sprue brushing diameter [mm]	18	Closing Force [kN]	220
Ratio-lenght/Diam screw	20	Distance between columns [mm]	254
Maximum injection volume [cm3]	20.4	Maximum mold thickness [mm]	400
Maximum weight / injection [g]	18.6	Maximum openning [mm]	200
Injection force [kN]	65.8	Minimum mold thickness [mm]	200
Injection rate [g/s]	38.4	Maximum mold weight [kg]	150
Maximum injection pressure [bar]	2587	Openning force [kN]	40
Maximum screw stroke[mm]	80	Closing force [kN]	17.6
Nozzle contact force [kN]	48	Ejection course [mm]	80
Back pressure stroke [mm]	180	Ejection force; forward/backward [kN]	18.1-12
Screw torque[Nm]	1802	Hydraulic force [bar]	160
Screw Velocity [U/min]	500-4002	Oil tank capacity [litrers]	115
Back pressure force [kN]	45.7	Work power [kW]	5,5
Heating capacity [W]	3250		
Hopper size [litre]	13		

The injection parameters used in the injection process are reported in Table 4- 4, as C1 and C7 are used as references for comparison;

Table 4- 4 Injection parameters used for each mixture

Composition	Designation	IP (bar)	IV (mm/s)	2IP (bar)	HT (s)	IT (°C)				
						1	2	3	4	5
C1	PP$_{100}$	115	120	90	4	150	170	176	180	180
C2	PP70GTR(0.25mm)30	90	120	35	3	150	170	170	180	180
C3	PP50GTR(0.25mm)50	110	120	35	3	150	170	170	180	180
C4	PP30GTR(0.25mm)70	120	140	45	3	150	170	170	180	180
C5	PP70GTR(1mm)30	110	120	45	3	150	170	170	180	180
C6	PP50GTR(1mm)50	110	120	45	3	150	170	170	180	180
C7	TPE (IRHD 15:43)	70	120	35	3	150	170	170	180	180

Where: IP is the Injection Pressure, IV is the Injection Velocity, 2IP is the second Injection Pressure, HT is the Holding Time, IT is the Injection temperature.

As can be seen from Table 4- 4, some parameters differ from a mixture to another, this is due to a troubleshooting that was done after bad results as; not filling the full cavities and sinking in some regions of the parts. This can be explained as for high content of rubber the viscosity of the blend increases considerably hence there is a need for more injection pressure and/or velocity.

b) **Mould:** The mould used was machined from a standard structure 196x156, supplied by BruYRubio. The two cavities mould (Figure 4- 8), designed in accordance with standards of EN ISO 527-4 [79], has a cold runner with side gate injection composed of two cavities with a standard cylindric ejection.

Figure 4- 8 Mould used for tensile samples

5. Characterization Techniques and Tests

5.1. Hardness Test

5.1.1. Shore (Durometer) Hardness

A durometer is a device that is built to determine the hardness of a material, whether the material is polymeric or elastomeric. The hardness of the material measured using this instrument is defined by a force that is applied by a typically designed pressure foot; the force applied on the material measures the depth of indentation on the material. In general, the hardness of the material will be calculated by the depth of indentation. In shore hardness test or durometer hardness test different materials are measured using different hardness scales [82]. According to American Society for Testing and Materials (ASTM D2240-00) uses twelve scales depending upon the type of usage. The twelve types of scales are denoted with letters A, B, C, D, DO, E, M, O, OO, OOO, OOO-S and R. Each of these scales range from values 0 to 100 as the hardness of the material increases [83]. The most used scales in in polymers are type A and type D. Type "A" scale is used to test soft plastic materials while type "D" is used for harder ones and as the hardness increase or decrease different levels of scales are used [84], [85]. Figure 5- 1 shows the existing difference between the shore A and D are on the indenter tip.

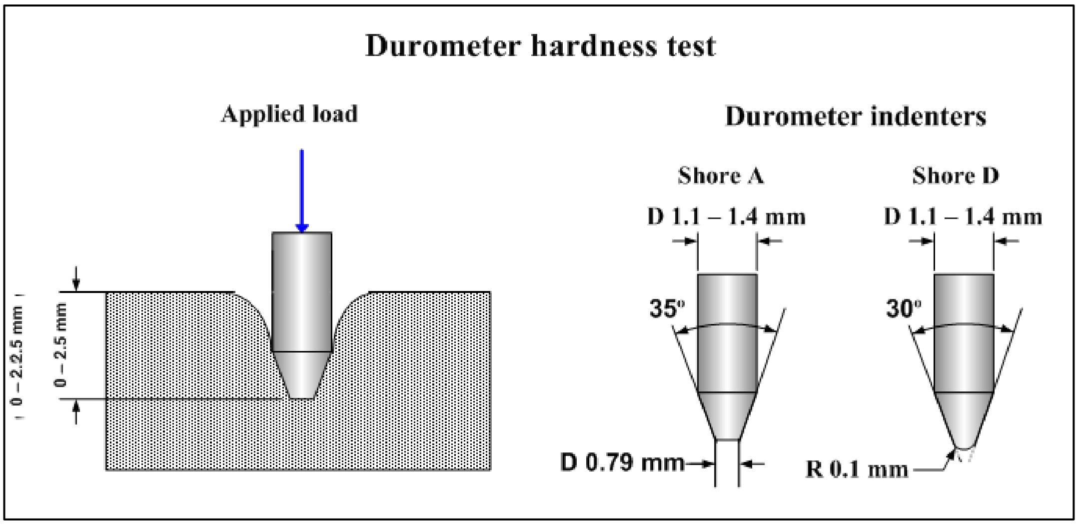


Figure 5- 1 Diagram of a durometer indenter or presser foot used for Shores A and D [82]

Figure 5- 2 exemplifies three types of hardness scale; Shore A, Shore D and Rockwell and shows the usual use of each one depending on the material family.

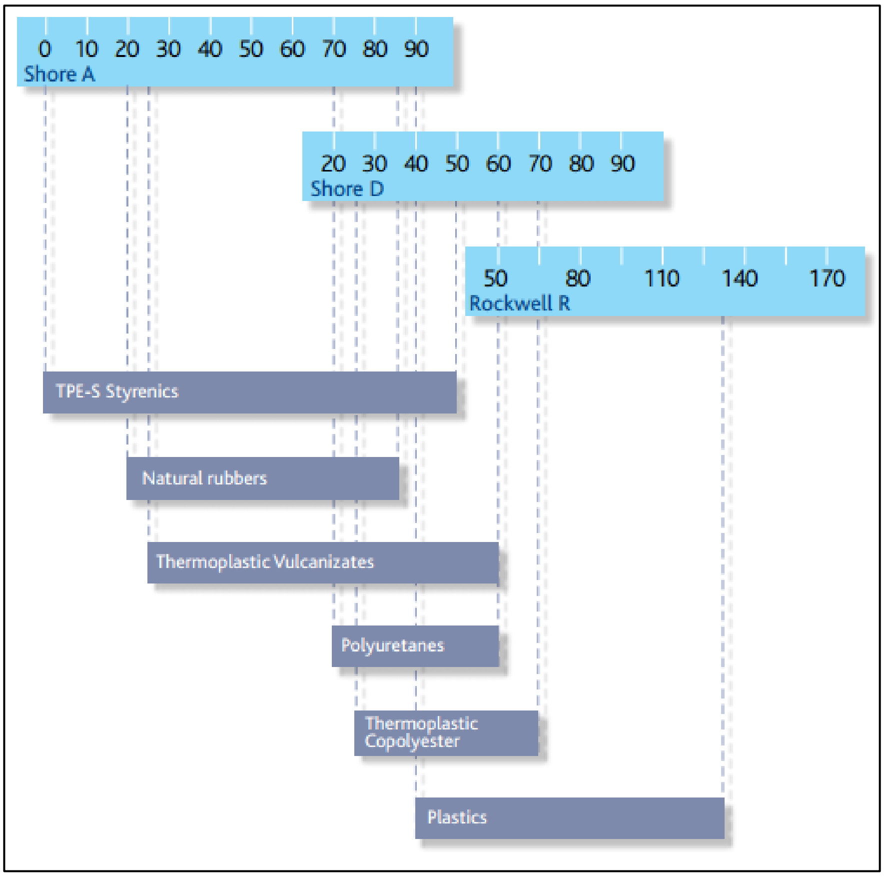


Figure 5- 2 Thermoplastic hardness range[65]

5.1.2. Test Procedure (ISO 868)

Since Shore A is usual scale for measuring the hardness of TPEs, so as a first test the samples were subjected to a durometer of Shore A. The results obtained were more than 80 and close to 90 for some. Another test with Shore D scale was performed by taking into consideration ISO 868 clause 8.2; that specifies whenever readings on shore A exceeds 90 shore D should be used instead, and whenever readings on shore D are less than 20 shore A should be used.

On this experiment, the shore A test was performed in *quality control laboratory of PLIMAT - Plastics Industrial Matos, SA,* and the shore D test was done in mechanical laboratory of ESTG. Figure 5- 3 shows the equipment used for the shore D test with the following conditions:

- The standard followed for was ISO 868 for Shore A and D,
- The reading time starts when the point of the rod enters in contact with the sample's surface for this a digital chronometer with a sound alarm was used to indicate the end of the interval,

- Reading/sample: number readings in different location (spaced at least with 6) for the same sample, the results was averaged and displayed as a single value for each sample,
- Samples/mixture: number of samples for each different material mixture.

The results are displayed according to ISO 868; it can be found **D/15:45** means that the results is **45** on shore **D** obtained after **15** seconds following the first contact of the durometer rod and the sample surface.

Table 5- 1 Shore Test conditions

Standard Ref.	ISO 868
Sample Ref.	same in ISO 527-2
Storage Environement	
Temp °C	25
Humdity %	75
Time h	>16
Testing Environement	
Temp °C	25
Humdity %	75
Reading Time s	15
Readings/Sample	6
Samples/mixture	3

.

Figure 5- 3 Shore D test equipment

5.2. Contact Angle (wettability) Test

Contact angle test is basically used to determine the wettability of a material. The wettability of a material is determined by measuring the contact angle; the contact angle is formed when the liquid comes in contact with the interface of solid or liquid. If the contact angle is smaller, the wetting property is larger (i.e. the material is said to have a wettability property if the contact angle between the liquid and solid interface is smaller than 90° and the material is said to have a non-wettability property if the contact angle between the liquid and solid is between 90° and 180° [84].

The contact angle is geometrically calculated by applying a tangent line from the contact point along the liquid-vapour interface in the droplet profile as shown in Figure 5-4.

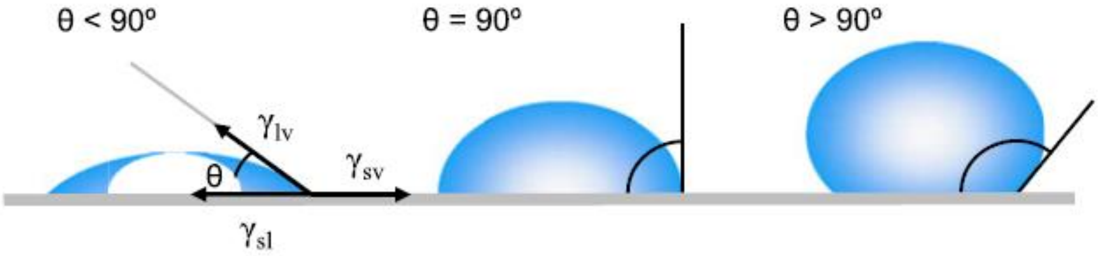


Figure 5- 4 Illustration of contact angles formed by sessile liquid drops on a smooth homogeneous [36]

As Thomas Young [15] in 1805 reported, the contact angle of a liquid drop on an ideal solid surface is defined by the mechanical equilibrium of the drop under the action of three interfacial tensions. It is represented by, what usually referred to, Young's equation [84];

$$\gamma_{lv} \cos \theta_Y = \gamma_{sv} - \gamma_{sl}$$

where γ_{lv}, γ_{sv}, and γ_{sl} represent the liquid-vapour, solid-vapour, and solid-liquid interfacial tensions, respectively, and θ_Y is the contact angle, or Young's contact angle [84].

Figure 5- 4 shows that the value of reference is 90° for contact which would be interpreted as incomplete wetting of the material and would be called in this case as a hydrophilic material, and much less than 90° would be correct to say that the surface of the material is favourable for wetting, while the contact angle is more than 90° generally means that the wetting of the surface is unfavourable which in this case would be called as a hydrophobic material. When the contact angle is greater than 150°, the surface is considered as a super-hydrophobic [84].

Lately, the wetting property has gained tremendous, as it is an important feature in many industrial processes, such as oil recovery, lubrication, liquid coating, printing, and spray quenching [53],[84].

The contact angle test was performed in CDRSP-IPLeiria using an ATTENSION THETA equipment (Figure 5- 5).

Figure 5- 5 ATTENSION THETA used for contact angle test

5.3. Tensile test (ISO 527)

ISO 527 is a standard that is framed to determine the tensile properties of composites and plastics under specified conditions. This standard explains various types of test specimens and methods to investigate the tensile behaviour of different materials. This standard explains that the tensile behaviour is determined by extending the specimen along the major longitudinal axis by applying constant load on the specimen until the fracture limit or until the stress or strain reaches the nominal value. The result is calculated on the basis of load sustained by the specimen and the amount of elongation [86].

Why it is necessary to perform a tensile test? In fact, it is very interesting to know how much load a material can bare. Usually tensile strength is performed in a universal testing machine and a graph is plotted as the specimen is stretched. The graph contains a curve that clearly explains the behaviour of the specimen under different load conditions. As the material is subjected to elongation at one point of load the material reaches the plastic deformation state and this point is termed as yield point. The last point where the specimen loses its strength and breaks is the ultimate strength of the specimen and this is the point where the specimen sustains the maximum load [87].

- **Stress-Strain Curves:** From the machine test, the tensile force is recorded as a function of the increase in gage length. Figure 5- 6a shows a typical force-elongation curve for a ductile material. While Figure 5- 6b shows a typical engineering stress-strain curve which is identical in shape to the first one. The engineering stress, or nominal stress, *s*, is defined as [87];

$$s = \frac{F}{A0}$$

where *F* is the tensile force and *A*0 is the initial cross-sectional area of the gage section. And The engineering strain, or nominal strain, *e*, is given as [87];

$$e = \frac{\Delta L}{L0}$$

where *L*0 is the initial gage length and Δ*L* is the change in gage length (*L* - *L*0). Stress strain curve is used to test the loading capacity of materials. The behaviour of the material as it is pulled apart until the specimen loses its strength is recorded in stress strain curve. Stress strain curve gives a clear visibility about the ranges of yield point, limit of plastic deformation, maximum tensile strength and the fracture limit. The values shown in the stress strain curves are very important for engineers to know the maximum limit of force that a material can withstand with respect to the cross section [86].

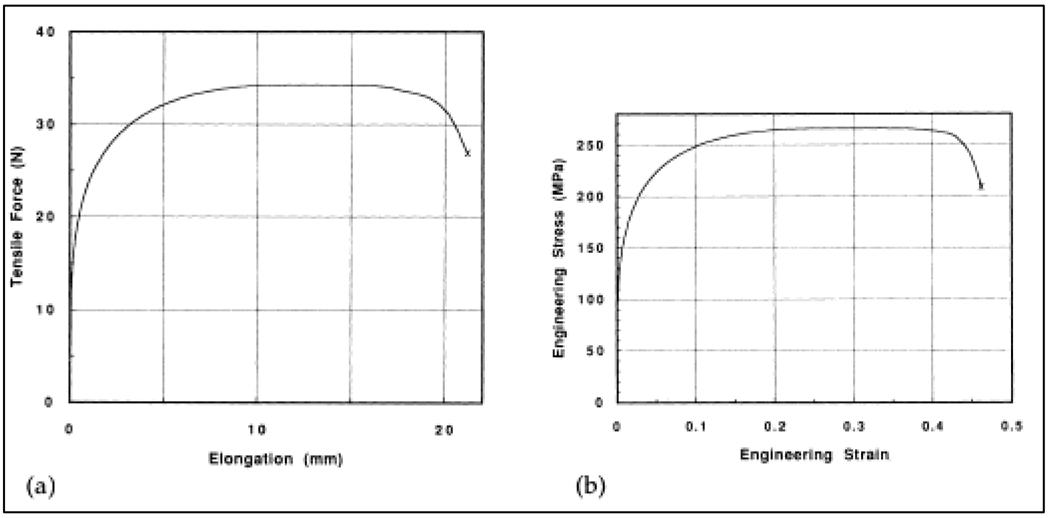


Figure 5- 6 (a) Load-elongation curve from a tensile test and (b) corresponding engineering stress-strain curve. Specimen diameter, 12.5 [68]

- **Elastic versus Plastic Deformation:** When the material is subjected to elongation, the first phase of damage to the specimen starts in the phase of elastic deformation. But in this stage, as the stress is removed the material goes back to its original shape. This is known as complete recovery or resilience [87]. When more stress is induced on the specimen after elastic phase, the material does not go back to its original shape this means the specimen has reached the plastic limit. This is the stage where the material has reached its yield value and the originality of the material cannot be retained further [87]. Those stages are depicted in Figure 5- 7, where A is the yield point, D is the ultimate tensile strength and F is the fracture point.

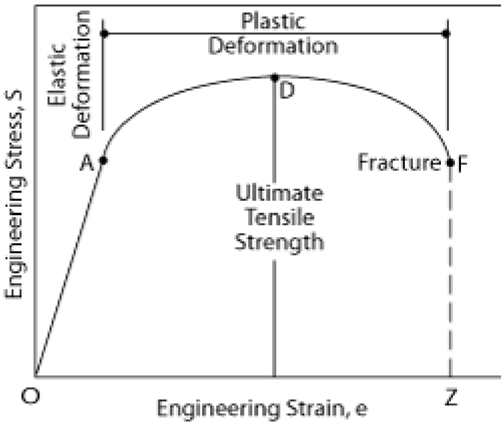


Figure 5- 7 Engineering stress-strain curve showing elastic and plastic deformation [88]

For most materials, the initial portion of the curve is linear. The slope of this linear region is called the *elastic modulus* or *Young's modulus, E:*

$$E = \frac{s}{e}$$

- **True Stress and Strain:** are used for accurate definition of plastic behaviour of ductile materials by considering the actual (instantaneous) dimensions [89].

  True stress, s', is the force (F) divided by the actual area (A) [89]:

$$s' = \frac{F}{A}$$

by simplifying the above equation;

$$s' = s * (e + 1)$$

True strain, e', is change in length with respect to the instant length [89]:

$$e' = \int_{L0}^{L} \frac{dL}{L}$$

by simplifying the above equation;

$$e' = \ln(e + 1)$$

- **Modulus of Toughness**: is the work done on a unit volume of material as a simple tensile force is gradually increased from zero to the value causing rupture is defined as the Modulus of Toughness. This may be calculated as the entire area under the true stress-strain curve from the origin to rupture [90]. Toughness of a material is its ability to absorb energy in the plastic range of the material [91]. The function is given by [92]:

$$Ut = \int_0^{st} eds$$

To calculate the Ut, a numerical integration is done. Using the trapezoidal rule which is given by:

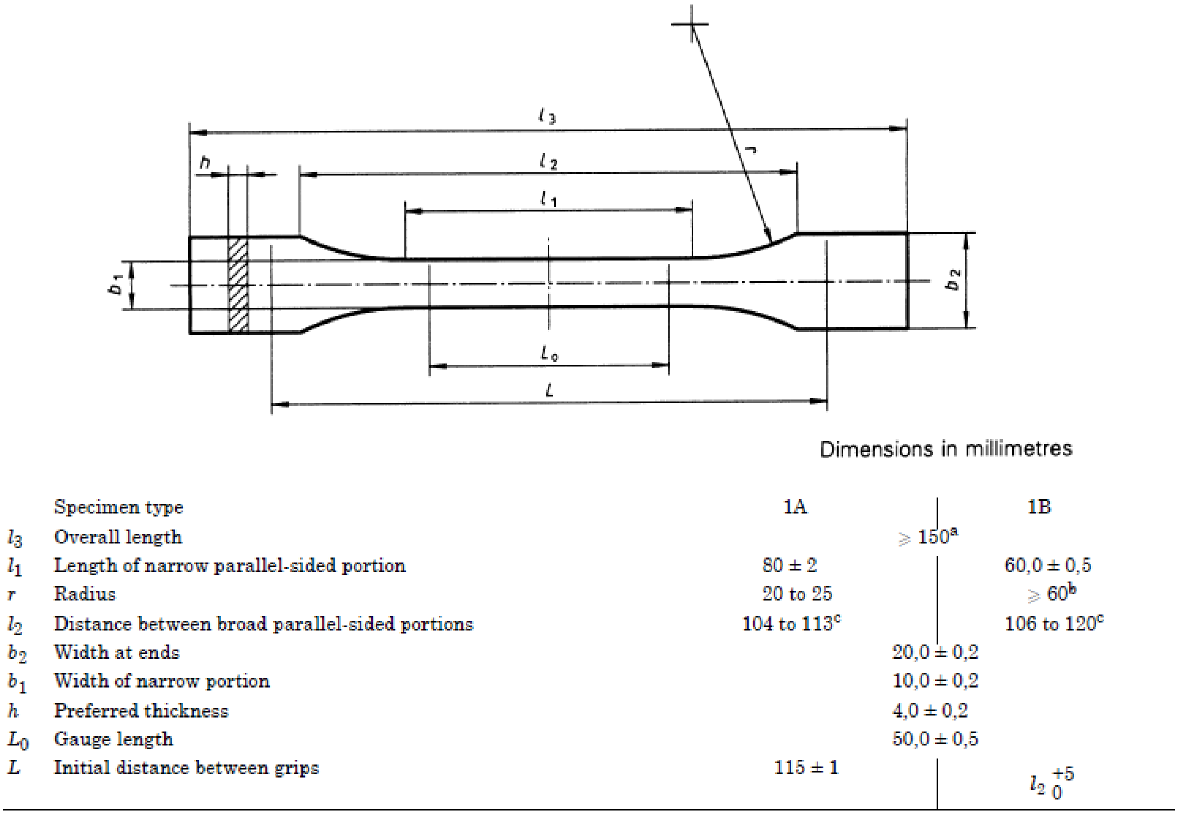
$$\text{Area}_0 = (Y_1+Y_0)*(X_1+X_0)/2$$

Where in this case; Y is the stress “e” and X is the strain “s” and Area_0 is the partial area between the pair (X_0, Y_0) and (X_1, Y_1).

Performing this formula in a recurring way for each successive values of stress and strain then summing all the results will generate the total area beneath the stress-strain curve(this was done using MS Excel), which is corresponding to the modulus of toughness. The unit is given by MJ/m^3.

5.3.1. Tensile specimens

Wherever possible, the test specimens shall be dumb-bell-shaped types 1A and 1B (ISO 527) , as shown in Figure 5- 8.



Dimensions in millimetres

	Specimen type	1A		1B
l_3	Overall length		⩾ 150[a]	
l_1	Length of narrow parallel-sided portion	80 ± 2		60,0 ± 0,5
r	Radius	20 to 25		⩾ 60[b]
l_2	Distance between broad parallel-sided portions	104 to 113[c]		106 to 120[c]
b_2	Width at ends		20,0 ± 0,2	
b_1	Width of narrow portion		10,0 ± 0,2	
h	Preferred thickness		4,0 ± 0,2	
L_0	Gauge length		50,0 ± 0,5	
L	Initial distance between grips	115 ± 1		$l_2\,{}^{+5}_{\ 0}$

Figure 5- 8 Test specimen types 1A and 1B [72]

The tensile test specimens should follow the standards of EN ISO 527-1. This standard explains that the test specimen should be free of twists; the edges must be free scratches, pits, and marks etc. Before the specimens are tested it should be checked for conformities by visual observation for straight edges, flat plates etc. The specimens should have enough space for gripping and testing. Minimum and maximum values are to be recorded for each specimen as specified in the standard EN ISO 527-1 and also should make sure that the specimens are within the tolerance values [87].

5.3.2. Tensile machine

Universal testing machine is the most commonly used for testing tensile, compression and bending. Universal testing provides results in a graph with stress on the Y- axis and strain on the X axis and hence, it is called stress-strain curve. These machines are either electromechanical or hydraulic. The main difference is the way the load is applied. Electromechanical machines are based on a variable-speed electric motor. It consists of a gear system that helps the cross head move up and down, by this the load can be applied on the specimen either in tension or in compression. The speed of the cross head can be changed by controlling the speed of the motor. Hydraulic universal testing machines consist of a hydraulic ram that moves the cross head up and down. The load is applied by adjusting the pressure of the orifice [87].

In this project, the samples were tested in electromechanical machine from Universal Instron model 4505 located in the research centre CDRSP, illustrated in Figure 5- 9.

Figure 5- 9 Universal Instron model 4505 used for tensile test

5.4. Morphology Test

Micro-CT (micro–computed tomography) is x-ray imaging in 3D, by the same method used in hospital CT (or "CAT") scans, but on a small scale with massively increased resolution [93].

X-ray microtomography, like tomography and x-ray computed tomography, uses x-rays to create cross-sections of a physical object that can be used to recreate a virtual model (3D model) without destroying the original object [94], [95].

The applications of micro-CT exceed medical imaging as it has reached industry. Generally, there are two types of scanner arrangements. The first is, the X-ray source and detector are stationary during the scan while the sample rotates. While the second is, while the specimen is stationary in space, the X-ray tube and detector rotate around. These scanners are typically used for small specimens (in some cases animals), biomedical samples, foods, microfossils, and other studies for which minute detail is desired [96].

The X-ray microtomography system was first developed and built by Jim Elliott in the early 1980s [97].

Material used: For this test, as the aim was to check the internal structure of samples, a SkyScan (1174v2) from Bruker, Figure 5- 10, was used with the following parameters;

- Voltage: 50 kV,
- Current: 800 µA,
- Pixel size: 11.23 µm,
- Execution time: 3500 ms,
- Rotation: 0.700°,
- Without any filter

Figure 5- 10 SkyScan-Bruker used for micro CT test [78]

6. Results and Discussion

6.1. Shore Hardness Test

The tests were performed under the conditions specified previously, the results were entered in an excel spreadsheet to calculate the average for each readings/sample (C1, C2, and so on...), the average and the standard deviation (Std) for the three samples.

Table 5- 2 Shore D hardness values of different mixtures

Composition	Mixture	SP1	SP2	SP3	Average	Std
C1	PP 100	D/15:54	D/15:53	D/15:52	D/15:53	0,92
C2	PP70/GTR(0.25mm) 30	D/15:53	D/15:54	D/15:53	D/15:53	0,38
C3	PP50/GTR(0.25mm) 50	D/15:47	D/15:47	D/15:48	D/15:47	0,38
C4	PP30/GTR(0.25mm) 70	D/15:36	D/15:37	D/15:37	D/15:37	0,72
C5	PP70/GTR(1mm) 30	D/15:53	D/15:50	D/15:53	D/15:53	1,69
C6	PP50/GTR(1mm) 50	D/15:49	D/15:50	D/15:50	D/15:50	0,58
C7	TPE (measured on IRHD scale)	IRHD 15:44	IRHD 15:43	IRHD 15:43	IRHD 15:43	0,64

Figure 5- 11 Mean of hardness values from different mixtures

From the data reported in Table 5- 1 and Figure 5- 11, it can be seen that adding 30wt% of GTR, regardless to particle size, does not affect the hardness of the mixtures as the values stayed the same of pure PP, D/15:53. In fact, the results have shown that adding smaller amounts of GTR to PP (30wt%) does not have an influence in the hardness obtained (Samples C1, C2 and C3). Moreover, when comparing samples C2 and C5 with different GTR particle sizes, the results continue to be equal. Probably, owing to the good dispersion of the particles of GTR in the PP matrix making possible to the durometer's tip to hit mainly the PP matrix due to geometric conditions.

For 50wt% of GTR, a drop of 5.7% for 1mm particle size and of 11.3% for the 0.25mm particles were registered during the test. This also can be assigned to the influence of GTR particles size and distribution due to the presence of higher percentage of GTR in the mixture.

The hardness value decreases even further with increase in the 0.25mm particles content for 70wt% to a value of 37 on shore D, which represent 32.3% less than the pure PP. Because of big percentages of GTR, now the hardness is mainly influenced by GTR and not PP as seen earlier.

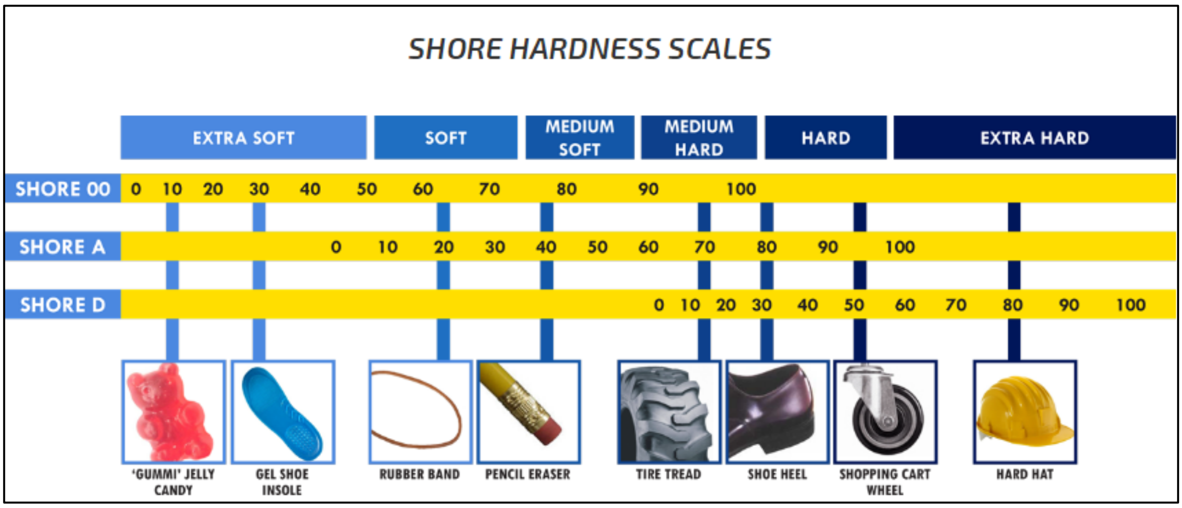


Figure 5- 12 Shore A-D hardness scale cross references [35]

Sukanya *et al.* [98], studied the properties of thermoplastic elastomers from waste polyethylene (WPE) and reclaim rubber (RR). The study covered different percentages on the mixtures (0,5,10,15,20,30…,70wt% of RR), they observed that the shore D hardness increase as the content increase up until 15wt% then it was a dead band until they pass the 30wt%.

In the present case, the dead band was observed until 50wt% of GTR content were added, which can be interpreted as; absence of agglomerated GTR particles in the mixture and the lack of interaction with the PP particles. More experiments with different content percentages should be performed to evaluate the precise interval of the dead band for each particle size. Beside that it can be concluded that, finer particles have a better interaction with the PP matrix which make the resultant blend softer.

6.2. Contact Angle Test

The experiment was developed according to the method previously described, giving the results for the different specimens, which are displayed on Table 5- 3. The mean calculated from the experimental results is shown in Figure 5- 13.

Table 5- 3 Contact angles values for different mixtures

Compositions	Mixture	SP1	SP2	SP3	MEAN	STD
C1	PP 100	77,21	80,86	80,18	79,4	1,9
C2	PP70/GTR(0.25mm) 30	88,47	86,43	88,35	87,8	1,1
C3	PP50/GTR(0.25mm) 50	89,67	89,04	92,31	90,3	1,7
C4	PP30/GTR(0.25mm) 70	88,63	87,70	89,71	88,7	1,0
C5	PP70/GTR(1mm) 30	83,59	83,89	80,98	82,8	1,6
C6	PP50/GTR(1mm) 50	90,03	88,20	89,55	89,3	0,9
C7	TPE (IRHD 42)	98,06	102,34	102,87	101,1	2,6

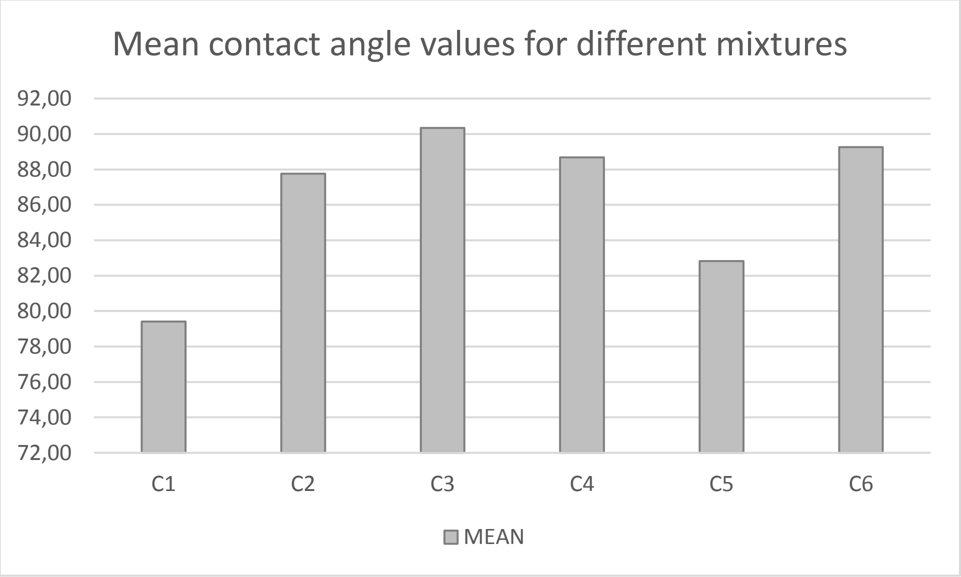


Figure 5- 13 Contact angle mean for different mixtures

The results shown in Table 5- 3 and Figure 5- 13, indicates that incorporating GTR particles with PP improves the wettability property as it increases for different percentages of GTR and as well for different particle sizes.

The contact angle increases 10.6% when adding 30wt% of 0.25mm particles and 4.3% when adding 30wt% of 1mm particles.

The maximum contact angle recorded for the blend PP/GTR is 90,3° for PP50GTR(0.25mm)50 which represents an increase of 13.7% when compared to pure PP. Thus, it was observed a slight decrease when adding 70wt% of GTR content compared to the 50wt%.

Hossain *et al* [99] investigated moisture damage resistance of GTR-modified asphalt binder by static contact angle measurements, and observed a continuous-decline trend of contact angle with an increase of GTR content.

In the present case; the results reported show the particle size have more influence on the wettability performance of the mixture than the particle content. This can be assigned to a better GTR/PP interaction.

This can be of great use when the wettability property is considered as an important feature on the material. Independently from the other properties and just taking into consideration the hydrophobicity, this mixture can be used for coating, outdoor use and electrical insulation.

6.3. Tensile Test

The prepared specimens were tested with the speed of 1mm/min as specified in ISO 527-2, clause 9. The mean of the results and their respective deviations, were calculated at least with 4 samples for each mixture, are depicted in Table 5- 4.

Table 5- 4 Tensile test result for different mixtures

MATERIAL	Tensile Strength (MPa)		Elastic Modulus (MPa)		Elongation at Break (%)		Modulus of toughness U_t (MJ/m3)	
	Mean	STD	Mean	STD	Mean	STD	Mean	STD
PP 100	21,83	--	427,12	--	35,00	--	18,07	--
PP70/GTR(0.25mm) 30	14,88	0,14	272,64	28,64	33,46	6,75	3,45	1,59
PP50/GTR(0.25mm) 50	10,52	0,30	189,57	5,93	28,24	2,89	2,41	0,30
PP30/GTR(0.25mm) 70	6,33	0,53	65,56	4,46	34,38	2,00	1,75	0,24
PP70/GTR(1mm) 30	13,98	0,37	290,92	14,02	18,30	2,21	1,80	0,32
PP50/GTR(1mm) 50	11,72	0,50	206,60	6,57	22,42	1,15	1,92	0,12

- **Tensile strength:**

From Table 5- 4, Figure 5- 14 and Figure 5- 15, can be seen that by increasing the content of GTR, independently from the particle size, the tensile strength decreases.

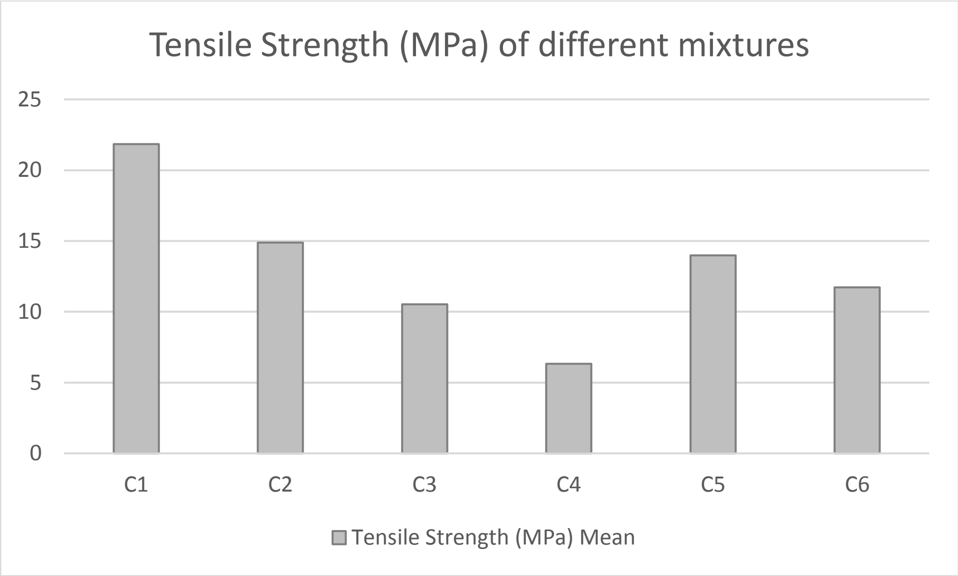


Figure 5- 14 Tensile strength of different mixtures

With GTR (1mm) the tensile strength decreases with 16.16% when the content increased from 30wt% to 50wt%. Comparing the mixtures having the same content of GTR but different particle size, with 30wt% those with 0.25mm particles shows higher values than those 1mm particles with 6%, and with 50wt% those with 0.25mm particles shows higher values than those of 0.25mm particles with 10.2%. In other words, particle size also has influence in the tensile strength, however with less impact the GTR content.

The same effect was overserved in many studies that included waste rubber tyres with different blends [100]–[102].

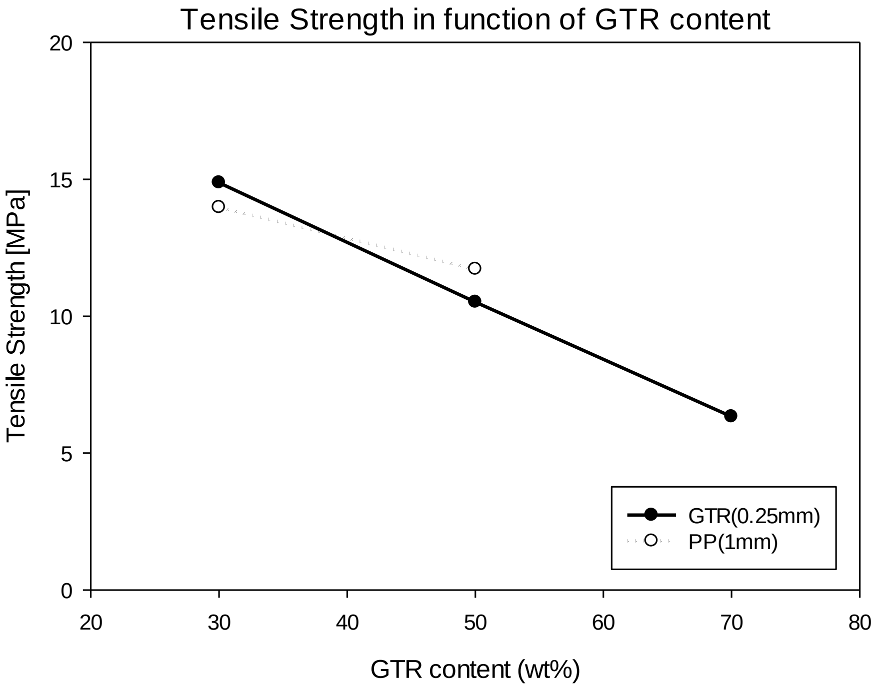


Figure 5- 15 Change in tensile strength values (MPa) in function of GTR content

- **Elastic Modulus:**

From Table 5- 4 and Figure 5- 16, it can be seen clearly that by adding GTR, whatever is the particle size, it makes the elastic modulus decrease gradually with the content of tyre rubber. Comparing the values of the mixtures with GTR, the 1mm particles shows higher values than the 0.25mm particles with 6.3% for 30wt% and with 8.2% for 50wt%. By adding 70wt% of 0.25m particles to PP, the elastic modulus decreases drastically with 84.7%.

The same effect was overserved in many studies that included waste rubber tyres with different blends [100]–[102].

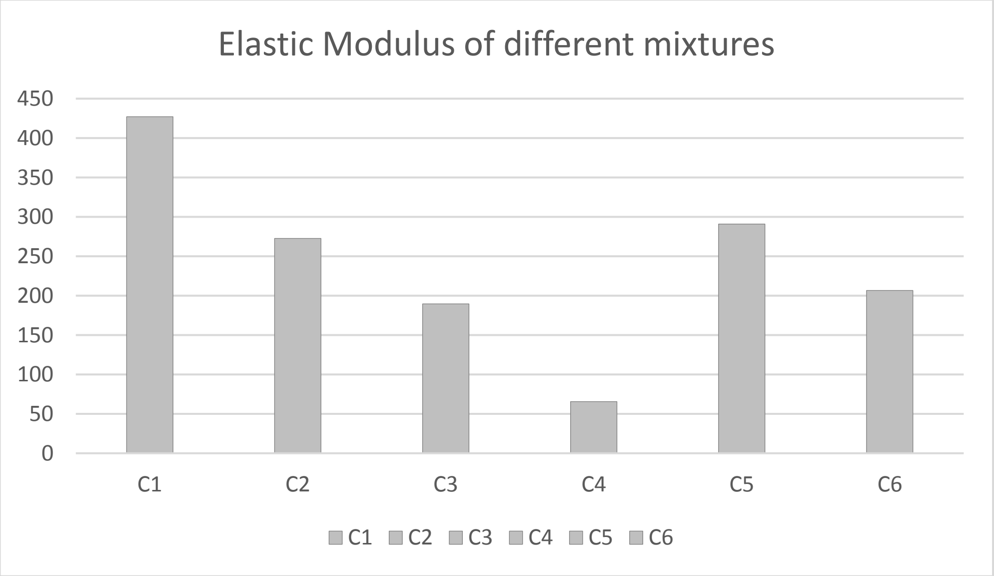


Figure 5- 16 Elastic modulus of different mixtures

- **Elongation at break:**

From Table 5- 4 and Figure 5- 17, adding GTR particles into PP induces the decrease on the elongation at break from the initial value.

The 0.25 mm particles showed higher values than the 1mm; for 30wt% of 0.25mm particles it was 33.46% and for 1mm particles was 18.23%.

The elongation at break of the 1mm particles increases when the content of GTR increases from 30wt% to 50wt%, which was not observed with the 0.25mm particles.
In fact, it was observed the minimum value for 0.25m particles when GTR is at 50wt%, Scaffaro *et al.* [15] observed the same tendency in their study of recycled polyethylene and GTR for both injection and compression moulding.
Goncharuk *et al.* [103] observed that elongation at break of LDPE/GTR blends decreased with increasing particle mean size, and Liu et al. [101] noted the same tendency in the case of PP/EPDM powder blends.
From Table 5- 4, it can be seen, in the case of 0.25mm particles the values of elongation at break decreases when the content increases from 30wt% to 50 wt% and increases when the content increases from 50wt% to 70 wt%. Egodage *et al.* [100] studied waste PP with GTR, observed that the elongation at break decreases with the increase of the GTR content from 0 to 40wt% and increases after the 50wt% of GTR, this change decrease-increase occurred while the tensile strength keeps decreasing. Liu et al. [101] and Goncharuk *et al.* [103] studied the effect of the waste tyre ground sizing and concluded that; the ambient ground powder and the GTR prepared with roller grinding show higher elongation at break.

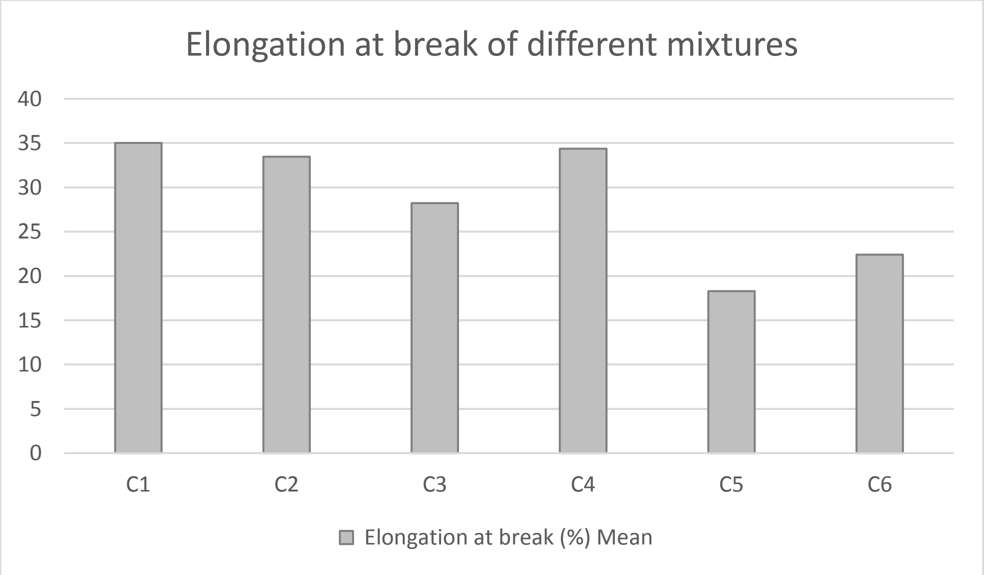


Figure 5- 17 Elongation at break (in %) of different mixtures

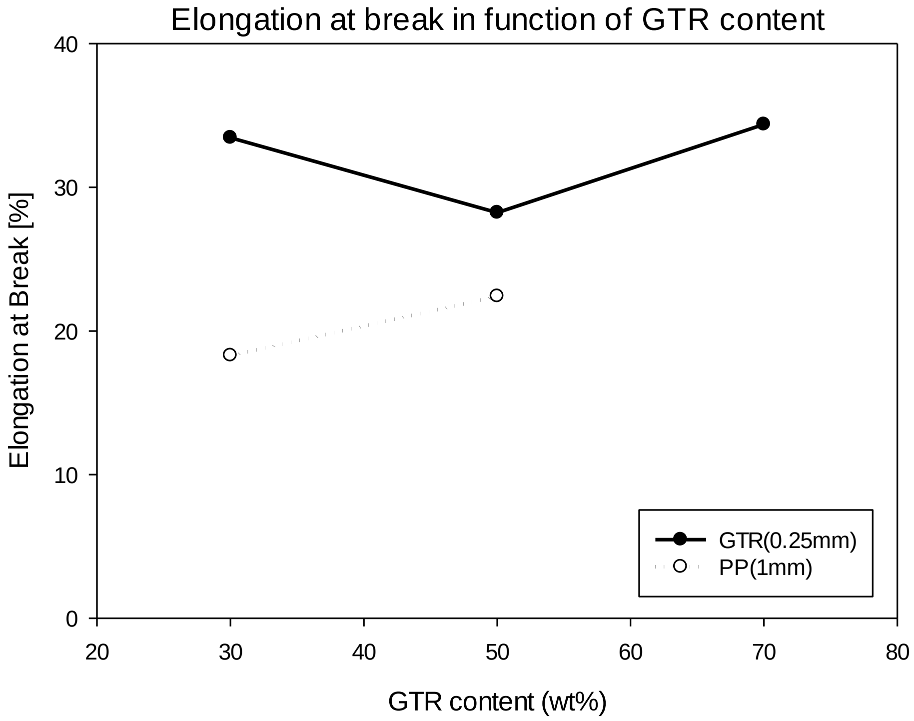


Figure 5- 18 Elongatio at break (%) in function of GTR content

Sukanya *et al.* [98] studied the properties of thermoplastic elastomers from waste polyethylene (WPE) and reclaim rubber (RR). It was observed that the tensile strength decreases and the elongation at break increases with the increase in the RR proportion in the blend. They explained this as; RR is a partially degraded rubber and contains carbon black as well as other additives in addition to the unperturbed sulphur crosslink [104].

Compared to the present work, the decrease of elongation at break when adding GTR can be explained by the type of rubber used is an unvulcanised GTR (even if some devulcanization occurred during the extrusion and the moulding).

- **Modulus of toughness:**

As can be seen in Table 5- 4 and Figure 5- 19, the modulus of toughness decreases as the content of GTR increases.

Independently from the particle sizes, the toughness decreases with a noticeable amount when compared to the value of pure PP.

With GTR (0.25mm) the modulus of toughness decreases with 80,9% when adding 30wt% of GTR. Increasing the content decreases even further the modulus of toughness to a final value of 1,75 for PP30GTR(0.25mm)70.

With GTR (1mm) the modulus of toughness decreases with 89,4% when adding 30wt% of GTR.

Egodage *et al.* [100], while studying waste polyethelene with ground tyre rubber (WPE/GTR), observed that as the GTR content increases the modulus of toughness decreases. Comparing with the values reported by Egodage *et al*. using WPE/GTR, the modulus of toughness' results from the present work using PP/GTR (Table 5- 4 and Figure 5- 19) are much higher.

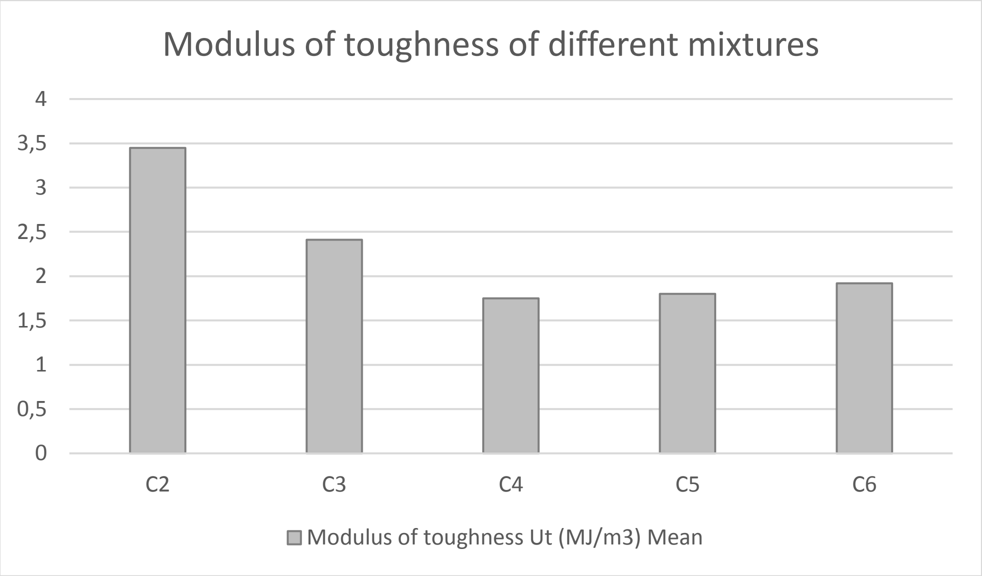


Figure 5- 19 Modulus of toughness of different mixtures

Strain versus stress curves are shown for different material compositions from Figure 5- 20 to Figure 5- 25.

.

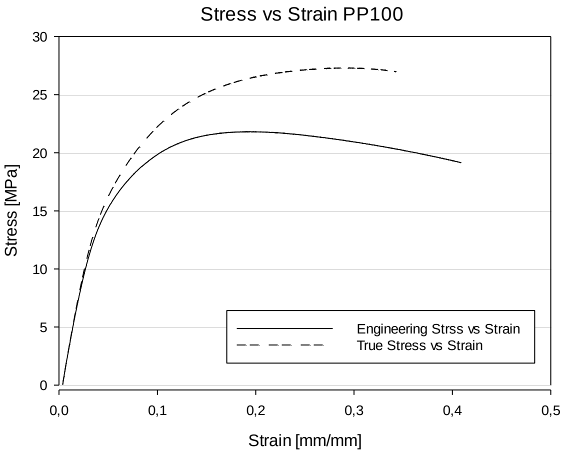


Figure 5- 20 stress vs strain for PP100 (pure PP)

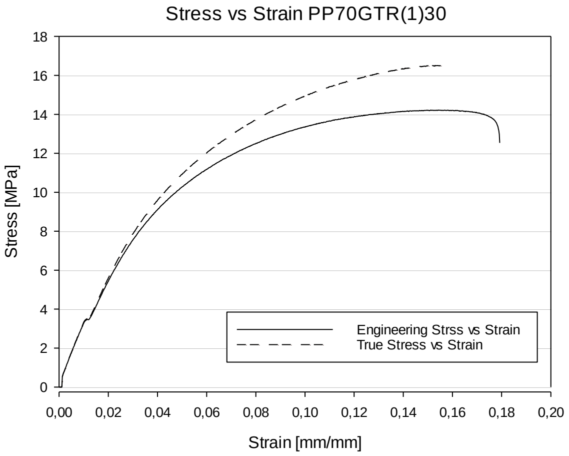


Figure 5- 21 Stress vs Strain for PP70GTR(1mm)30

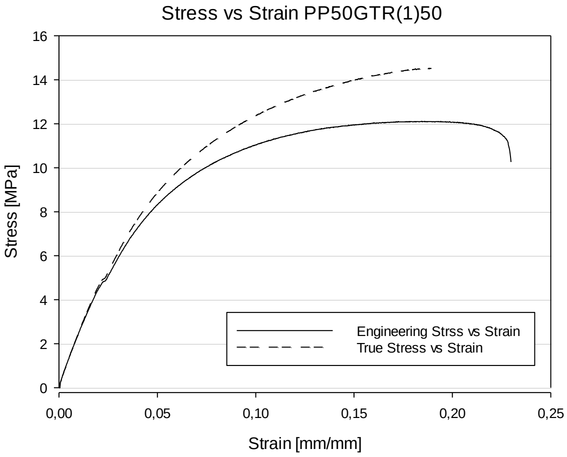


Figure 5- 22 Stress vs strain PP50GTR(1mm)50

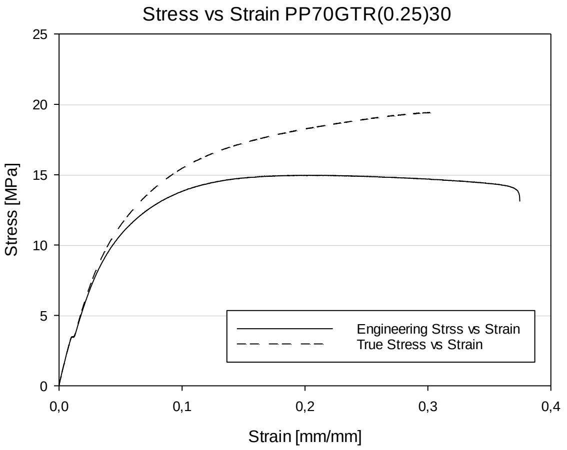


Figure 5- 23 Stress vs Strain PP70GTR(0.25mm)30

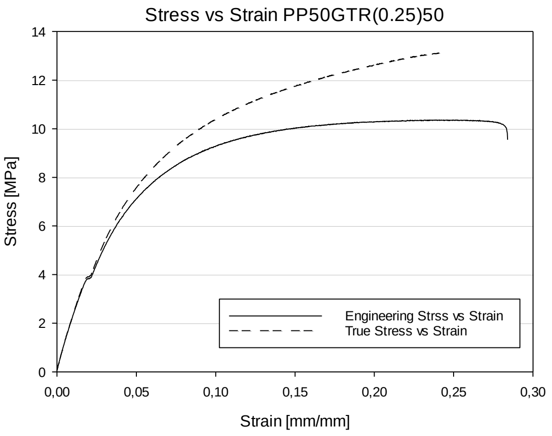


Figure 5- 24 Stress vs Strain PP50GTR(0.25mm)50

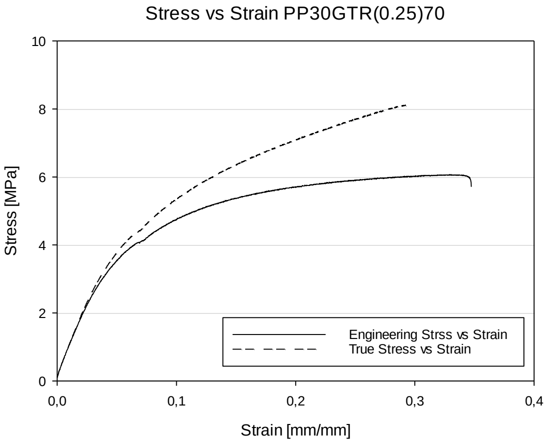


Figure 5- 25 Stress vs Strain PP30GTR(0.25mm)70

From the above curves; it can ba seen that only the curve of pure PP starts dropping before the fracture, which is not observed anymore as GTR particles is incorporated. It is noticebale that the inlceation of the different curves decreases as GTR is added (this is interpreted as the decrease of the elastic modulus). Beside to that, is the tensile strenght as the maximum value reached decreases as GTR content increases.

Figure 5- 26 shows a comparison between all the previous presented cases.

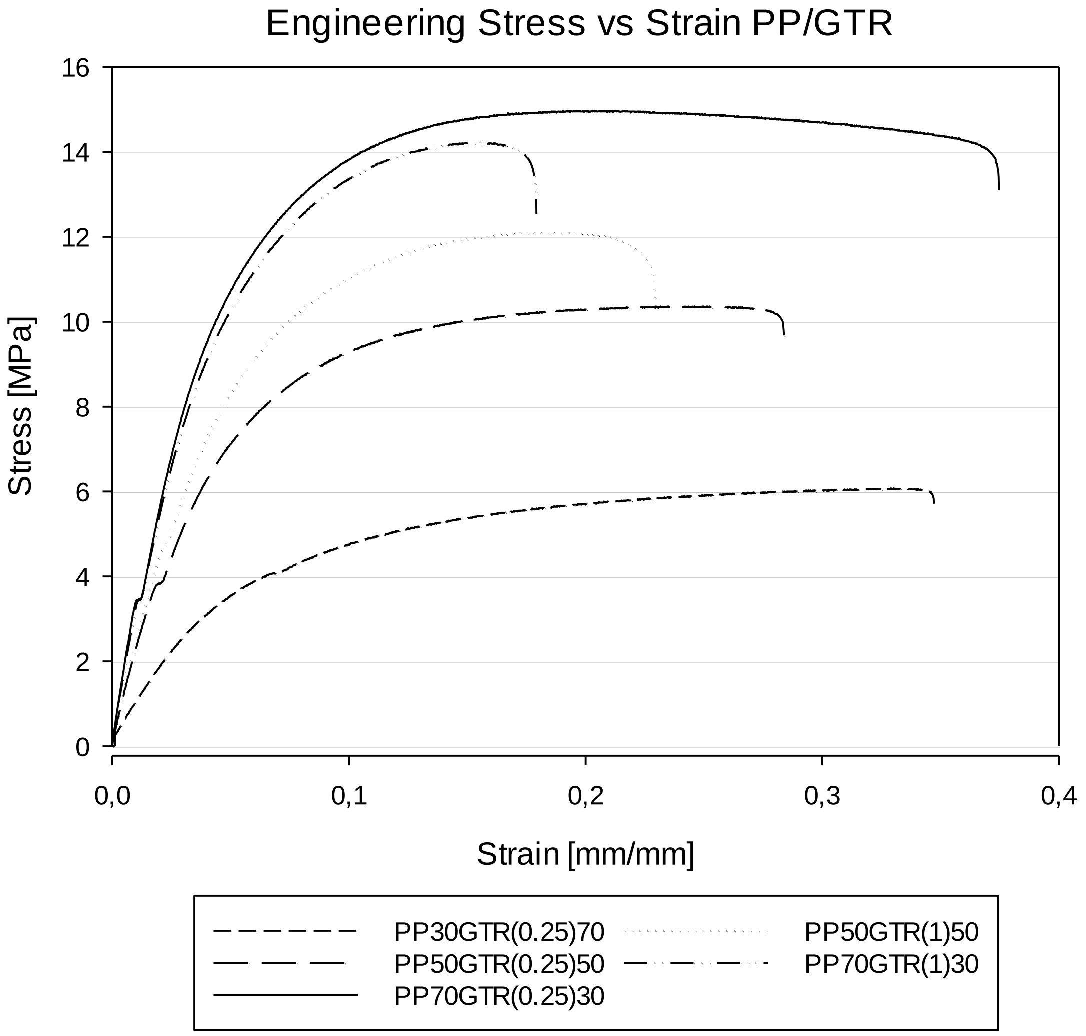


Figure 5- 26 Engineerng Stress-curves of PP/GTR with different content and particle sizes

As it can be concluded from analysing Figure 5- 26;

- The Elastic modulus is more influenced by the GTR content rather than the particle size. As by increasing the GTR content, the value of the Elastic Modulus decreases.
- Tensile strength, as can be seen, is influenced by particle size and GTR content.
- The particle size has more influence than the GTR content on the elongation at break as it was registered higher values for GTR(0.25mm),
- The modulus of toughness is highly influenced by the GTR particle size, as it was registered higher values for GTR(0.25mm).

6.3. Morphology Test

Using the equipment and technique discussed in chapter 4, and after data treatment, the results for each specimen are illustrated on the figures Figure 5- 27 to Figure 5- 37. To separate the matrix and the GTR particles, the micro-CT result were then treated on the software *ImageJ* an open source image processing program designed for scientific multidimensional images [105].

It can be clearly seen that the results obtained are not enough to see the interface between the blends, even though some conclusions can be made about the particles distribution, particles locations and samples porosity.

From the analysis of micro-CT result displayed on the following figures, it can be said that in all the mixtures there exist gaps between the phases. The bigger particle size the bigger is the gap, this can explain the decline mechanical properties when GTR is added.

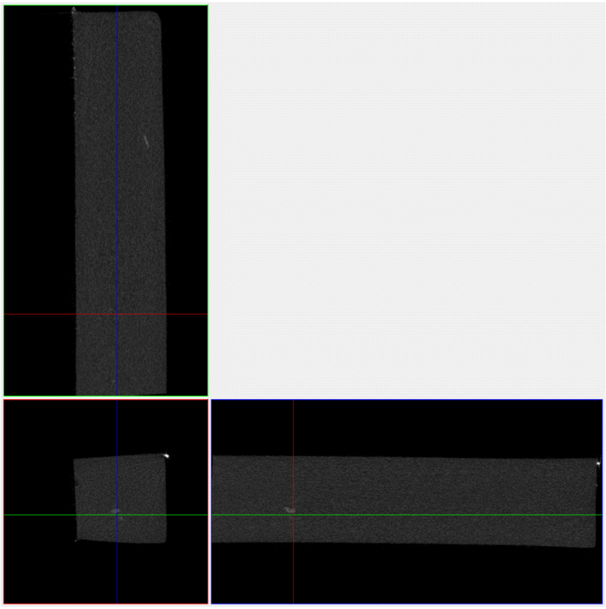


Figure 5- 27 micro-CT result for PP100

In Figure 5- 28 and Figure 5- 29, it can be said that the particles are not uniformly distributed on all surfaces and absence of isotropy. There exists some void point, air space spot, which can be assigned to the lack of mixture homogeneity, processing parameters (that probably led to some issues of compaction) and poor cooling system which must be tuned to provide better cooling process.

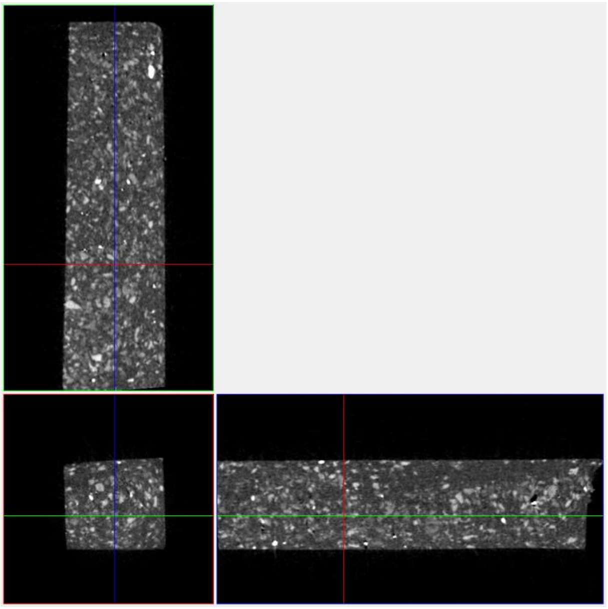


Figure 5- 28 micro-CT result for PP70 GTR(0.25mm) 30

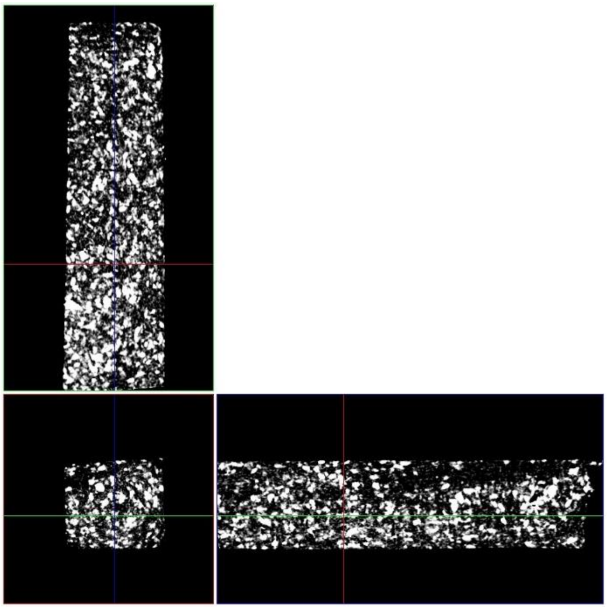


Figure 5- 29 ImageJ result for PP70 GT(0.25mm)30

From Figure 5- 30 and Figure 5- 31, it can be seen that the particles distribution is not balanced on the sample, and there are some spaces without GTR particles. This can be a reason for the decrease on the elongation at break registred on the tensile test.

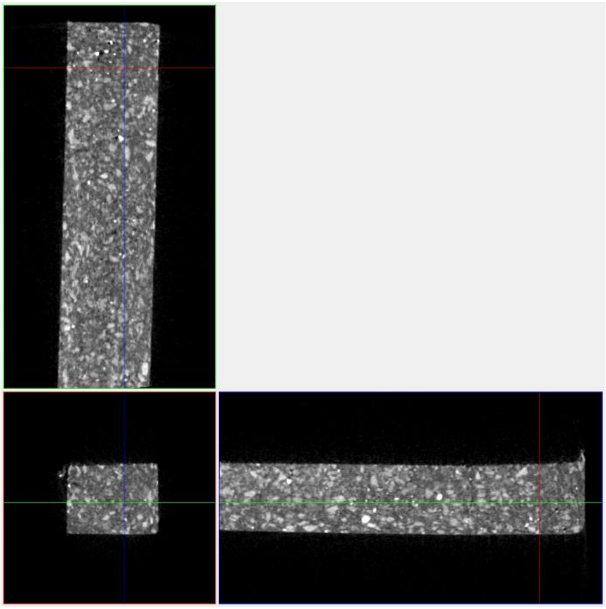


Figure 5- 30 micro-CT result for PP50 GTR(0.25mm)50

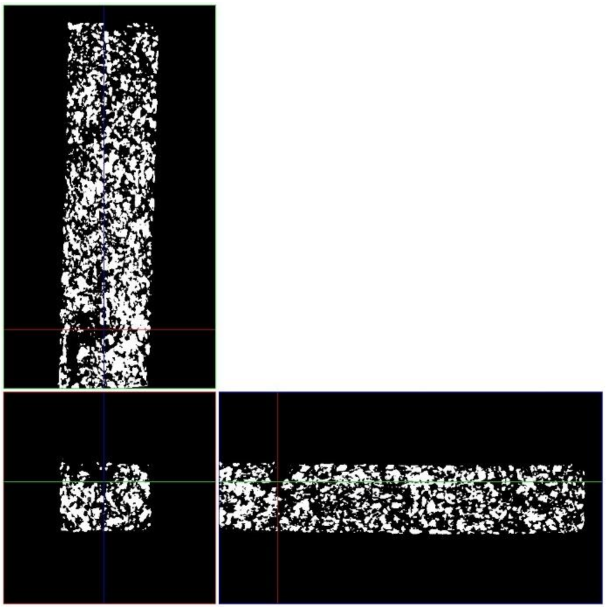


Figure 5- 31 ImageJ result for PP50 GTR(0.25mm)50

From Figure 5- 32 and Figure 5- 33, it can be seen that GTR particles are taking over the PP particles, this can explain decreasing results of shore D hardness.

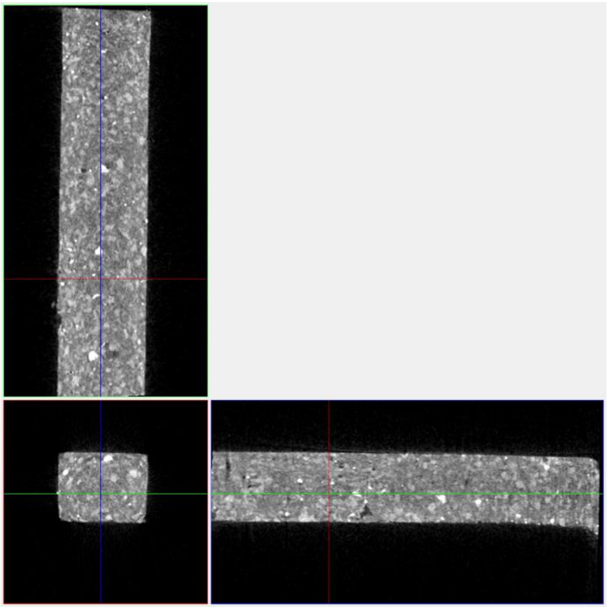


Figure 5- 32 micro-CT result for PP30 GTR(0.25mm)70

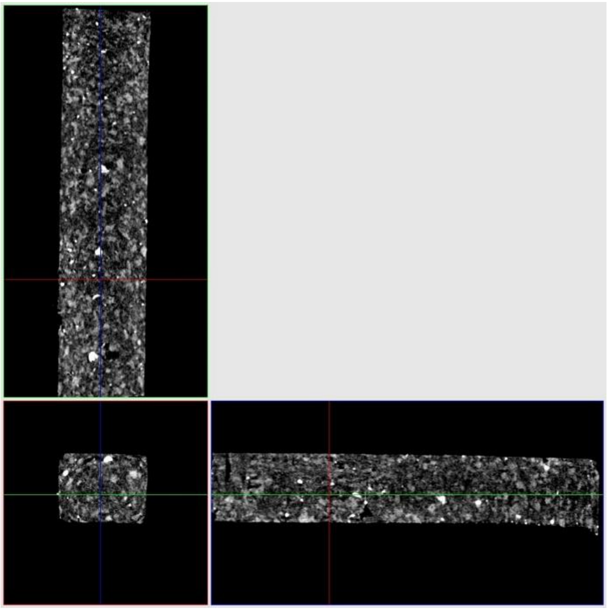


Figure 5- 33 ImageJ result for PP30 GTR(0.25mm)70

From Figure 5- 34 and Figure 5- 35, it can be seen the GTR particles of 2 mm, even with small percentage compared to 1mm, are taking big space on the samples. When compared to GTR(0.25mm), the distribution and the isotropy of GTR(1mm) is worst and the main reason can be assigned to the presence of 2mm particles.

The presence of 2mm particles can be the reason; of the processing difficulties encountered in the extrusion process (referred to in chapter 4) and probably also the reason for the poor performance (on elongation at break and modulus of toughness) already observed for this mixture.

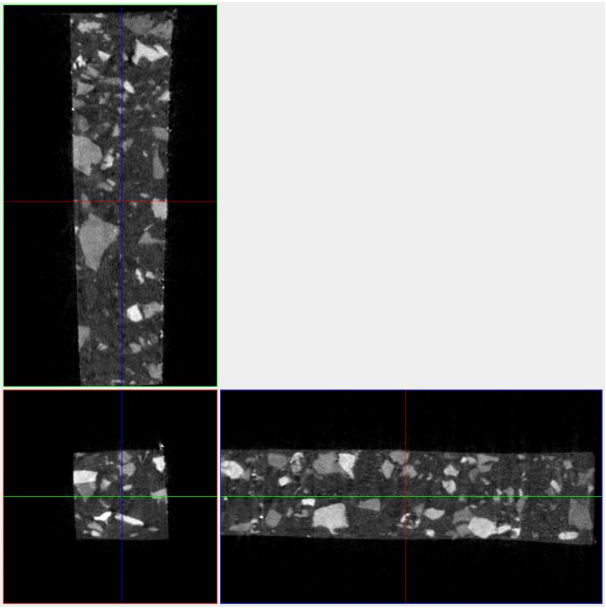


Figure 5- 34 micro-CT result for PP70 GTR(1mm)30

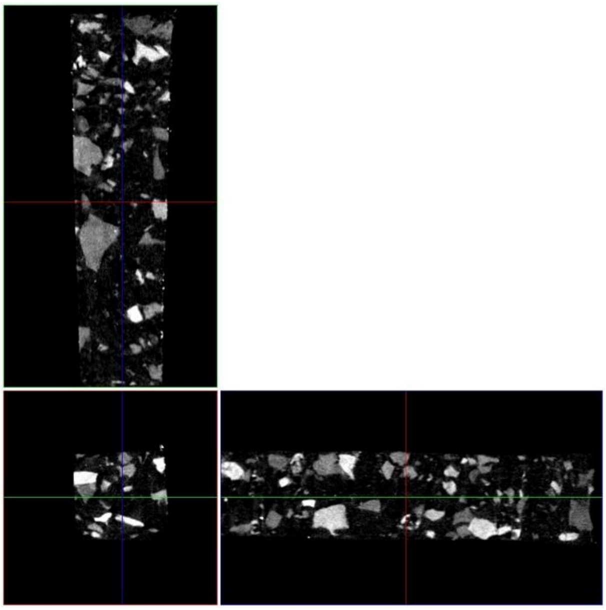


Figure 5- 35 ImageJ result for PP70GTR(1mm)30

From Figure 5- 36 and Figure 5- 37, the same situation as previously observed also occurs, the unbalanced distribution the GTR particles of 2 mm are compromising the structure of the sample. This latter could be the reason of the poor toughness results, compared with GTR(0.25mm), registered for both mixtures with GTR(1mm).

The concentration of the particles in one place can be notorious to the structure of the samples and the performance of the mixtures, as already cited in the previous results.

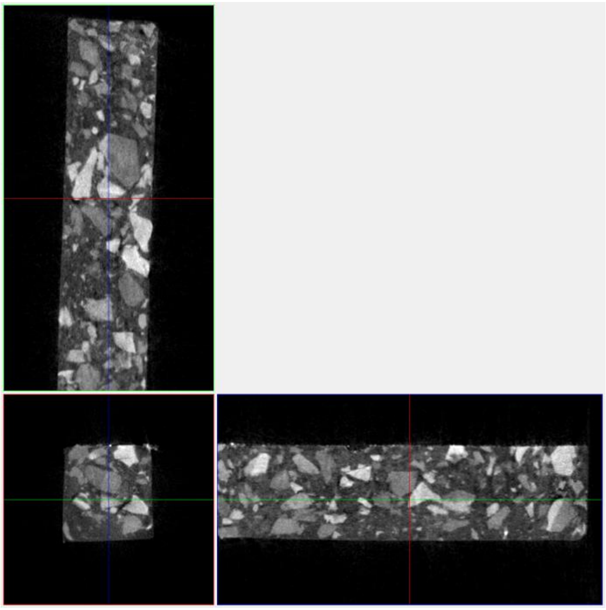


Figure 5- 36 micro-CT result for PP50 GTR(1mm)50

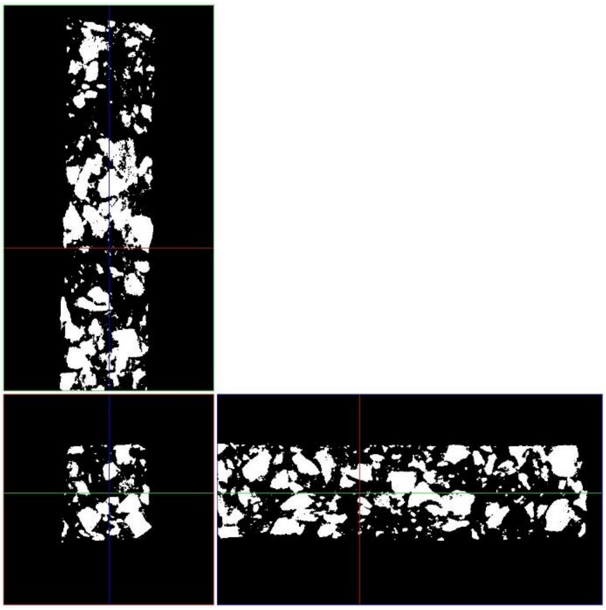


Figure 5- 37 ImageJ result for PP50 GTR(1mm)50

7. Conclusions

Compounding PP with GTR without using any additives or compatibilizer was carried out and the effect of; PP/GTR mixing ration and GTR particle size were considered.

It was observed during this study, that there exists a dead-band on hardness values. Regardless to the particle size, the values didn’t change only after adding 50wt% of GTR, similar behaviour already observed in other studies [98].

Adding GTR to PP enhanced the wetting property since the contact angles registered were bigger than the pure PP. This feature is well searched-for; since many investigations are working on developing cheap hydrophobic and super-hydrophobic materials.

It was observed that as the rubber concentration in the composite increases, the overall strength of the composite decreases. These observations are supported by the results reported by Huang *et al.* [106] and many other studies [63], [15], [100], [107]. This behaviour may be due to incompatibility between the hydrophobic rubber particles and the hydrophilic polypropylene that results in weak interfacial adhesion. This tendency worsens as the particle size mean increases.

When comparing the effect of particle size; compounding PP with GTR(0.25mm) showed a tougher composite, while the PP with GTR(1mm) resulted on a stronger one.

The GTR(0.25mm) showed better toughness, elongation at break and tensile strength for 30wt% than GTR(1mm). And GTR(1mm) expressed better elastic modulus.

For GTR(0.25mm) it was observed a decrease-increase on elongation at break, which Egodage *et al.* [100] already reported the same tendency. It is assumed to be due to unbalanced particle distribution as shown on morphology test.

Hence for the studied mechanical performances; the elongation at break and the modulus of toughness are influenced by the particle size, as for finer particles, it was observed higher values for both properties. While the elastic modulus and tensile strength are more influenced by the GTR content rather than the particle size, as for higher content it was reported smaller values.

The morphology outcomes backed the results of other tests, it was observed that; the PP/GTR blends are a composite-like mixture where GTR acts as a filler and PP as a matrix. The presence of gaps between the phases which increase as the particles size. These issues can greatly compromise the material performance which could explain the poor tensile results.

The morphology test showed that, even with small percentage, the presence of higher particle size (>1mm) on GTR(1mm) resulted on unbalanced distribution, increasing the gaps between the phases, which directly induced on brittle material (poor elongation at break and poor modulus of toughness).

After preforming the test previously cited and analysing their results. The composition PP/GTR was tried on additive manufacturing process, using a robot arm coupled with an extruder system and heated base (Figure 7- 1), available at CDRSP-IPLeiria.

Figure 7- 1 Robot arm used for Additive Manufacturing

The PP/GTR mixture was extruded, granulated and dried with the same parameters as C2 (referred on the previous chapters). A rotated-squared box was modelled and prepared for 3D printing (Figure 7- 2).

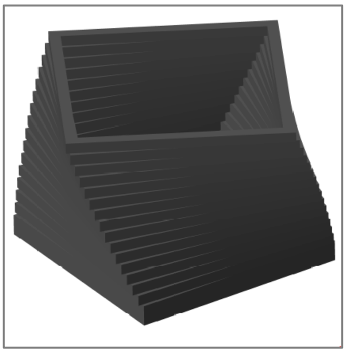


Figure 7- 2 3D model used for Additive Manufacturing

For the purpose of comparison, the additive manufacturing was done with two compositions; PP and PP/GTR. During the experiment the parameters used (temperatures of; the nozzle, the base and air-blown onto the printed layers) were subjected to tuning in the sake of achieving an acceptable result. Figure 7- 3 shows the final product of the process.

Figure 7- 3 Final result of Additive Manufacturing

Upon analysis of the result, the printed part showed;

- good adherence of the layers,
- better geometric results than other parts with different material,
- the problems encountered on the printed part were also present on others, hence those problems are not material related rather than process related,
- processing PP/GTR on additive manufacturing is a totally feasible technique. Which can be used for prototyping or producing complicated parts with scrap tire based material.

This study showed that Polypropylene and Ground Tyre Rubber can be used, without any additives added, to prepare polymer blends for injection moulding. This opens new insight for wider, and more industrialized, products' range as; safety road cones, safety road barriers, shoe heel, shopping cart wheel, electrical insulator.

These results should encourage engineers to design with PP/GTR in products (for outdoor use, coating, self-cleaning and among other usage). It is a way of promoting design for environment and design for recycling while preserving other features as cost and manufacturability since the material can be moulded by injection.

For reaching higher goals, some assignments can be addressed as future works mainly;

- Filtering GTR(1mm) to get rid-off all particles with size bigger than 1mm, and executing again the test to see the influence of performances of PP/GTR(1mm).
- More experiments with different content percentages should be performed to evaluate the interval, with a certain precision, of the dead band of hardness results for each particle size. Other particle sizes can also be added to the study.
- Checking other properties that were not covered by the present study as; impact strength, thermal and acoustic properties.
- For PP/GTR get better attention from users (engineers, producers and others), geometry and dimensional stability should be addressed properly, like that it will be checked if the PP/GTR fits for tight tolerances products.
- Injecting PP/GTR in another mould with venting, to prevent voids and burning caused by trapped air when the mould is closed. Preferably injecting a certain specific part (with holes, curves and parallel surfaces) enabling to check Geometric Dimensioning and Tolerancing (GD&T) properties.
- Optimizing additive manufacturing process and parameters.
- Characterizing and testing of products from additive manufacturing (as Delamination study)
- Use this material in different transformation processes (Rotomolding, Extrusion…)

References

[1] S. Murugan, M. C. Ramaswamy, and G. Nagarajan, “A comparative study on the performance, emission and combustion studies of a DI diesel engine using distilled tyre pyrolysis oil-diesel blends,” *Fuel*, vol. 87, no. 10–11, pp. 2111–2121, 2008.

[2] ETRMA, “End-of-life Tyre REPORT 2015,” 2015.

[3] ETRMA, “End of life tyres,” *Group*, 2006.

[4] J. D. Martínez, J. Rodríguez-Fernández, J. Sánchez-Valdepeñas, R. Murillo, and T. García, “Performance and emissions of an automotive diesel engine using a tire pyrolysis liquid blend,” *Fuel*, vol. 115, pp. 490–499, 2014.

[5] N. Fontaine and H. Védrine, “End of life vehicles,” in *Directive 2000/53/CE*, 2000, p. 18.

[6] European Commission, “End of Life Vehicles,” 2016. [Online]. Available: http://ec.europa.eu/environment/waste/elv/index.htm. [Accessed: 03-Sep-2017].

[7] O. Doğan, M. B. Elik, and B. Özdalyan, “The effect of tire derived fuel/diesel fuel blends utilization on diesel engine performance and emissions,” *Fuel*, vol. 95, pp. 340–346, 2012.

[8] S. Frigo, M. Seggiani, M. Puccini, and S. Vitolo, “Liquid fuel production from waste tyre pyrolysis and its utilisation in a Diesel engine,” *Fuel*, vol. 116, pp. 399–408, 2014.

[9] R. Alkhatib, “Development of an alternative fuel from waste of used tires by pyrolysis,” *MINES Nantes, l’U nam*, 2015.

[10] M. Sienkiewicz, J. Kucinska-lipka, H. Janik, and A. Balas, “Progress in used tyres management in the European Union : A review,” vol. 32, pp. 1742–1751, 2012.

[11] RMA, “Scrap Tire Publications,” 2017. [Online]. Available: https://rma.org/publications/scrap-tire-publications. [Accessed: 14-Dec-2016].

[12] JATMA, “TYRE INDUSTRY in Japan,” 2016.

[13] wbcsd, “End-of-Life Tires,” 2016. [Online]. Available: http://www.wbcsd.org/Projects/Tire-Industry-Project/End-of-Life-Tires. [Accessed: 14-Dec-2016].

[14] M. F. Ashby, *Materials Selection in Mechanical Design*. Elsevier, 2011.

[15] R. Scaffaro, N. T. Dintcheva, M. A. Nocilla, and F. P. La Mantia, "Formulation, characterization and optimization of the processing condition of blends of recycled polyethylene and ground tyre rubber: Mechanical and rheological analysis," *Polym. Degrad. Stab.*, vol. 90, no. 2 SPEC. ISS., pp. 281–287, Nov. 2005.

[16] A. Gent and J. Walter, "The pneumatic tire," no. February, 2005.

[17] T. Suparat, "Waste Tyre Management in Thailand : A Material Flow Analysis Approach," no. May 2013, 2013.

[18] Bridgestone Corporation, "Basic Structure-Radial Tires." [Online]. Available: http://www.bridgestone.com/products/speciality_tires/aircraft/products/basicstructure/radial.html. [Accessed: 21-Feb-2017].

[19] T. Amari, N. J. Themelis, and I. K. Wernick, "Resource recovery from used rubber tires," *Resour. Policy*, vol. 25, no. 3, pp. 179–188, 1999.

[20] D. Lo Presti, "Recycled Tyre Rubber Modified Bitumens for road asphalt mixtures: A literature review," *Construction and Building Materials*, vol. 49. Elsevier, pp. 863–881, 01-Dec-2013.

[21] Continental, "Tyre Basics: Passenger Car Tyres," *Tdc 06/2008*, pp. 1–30, 2013.

[22] H. L. H. J. and R. W. Eldredge, "Part 3: The Sanitary Landfill," *A Br. Hist. Solid Waste Manag. US Dur. Last 50 Years*.

[23] Ohio Environmental Protection Agency, "Regulations Municipal Solid Waste Landfills." [Online]. Available: http://www3.epa.gov/epawaste/nonhaz/municipal/landfill/msw_regs.htm. [Accessed: 30-Mar-2016].

[24] Environment Canada, *Technical Document on Municipal Solid Waste Organics Processing*. 2013.

[25] B. Adhikari and S. Maiti, "Reclamation and recycling of waste rubber," vol. 25, no. May, pp. 909–948, 2000.

[26] P. Ferrão, P. Ribeiro, and P. Silva, "A management system for end-of-life tyres : A Portuguese case study," vol. 28, pp. 604–614, 2008.

[27] DailyMail, "Worlds biggest tyre graveyard," *dailymail.co.uk*, 2013. [Online].

Available: http://www.dailymail.co.uk/news/article-2337351/Worlds-biggest-tyre-graveyard-Incredible-images-Kuwaiti-landfill-site-huge-seen-space.html. [Accessed: 05-Jun-2017].

[28] J.-W. Jang, T.-S. Yoo, J.-H. Oh, and I. Iwasaki, "Discarded tire recycling practices in the United States, Japan and Korea," *Resour. Conserv. Recycl.*, vol. 22, no. 1, pp. 1–14, 1998.

[29] "Monitoring a New Environmental Pollutant," *Anal. Chem.*, vol. 58, no. 1, p. 70A–74A, Jan. 1986.

[30] Steve, "The world's 9 worst tire fires." [Online]. Available: http://webecoist.momtastic.com/2013/05/28/burn-rubber-the-worlds-9-worst-tire-fires/. [Accessed: 11-Apr-2017].

[31] S. K. Ritter, "Tire Inferno," 2015. [Online]. Available: http://cen.acs.org/articles/91/i43/Tire-Inferno.html. [Accessed: 12-Aug-2017].

[32] K. E. Day, K. E. Holtze, J. L. Metcalfe-Smith, C. T. Bishop, and B. J. Dutka, "Toxicity of leachate from automobile tires to aquatic biota," *Chemosphere*, vol. 27, no. 4, pp. 665–675, Aug. 1993.

[33] J. J. Evans, "Rubber tire leachates in the aquatic environment.," *Rev. Environ. Contam. Toxicol.*, vol. 151, pp. 67–115, 1997.

[34] S. Ramarad, M. Khalid, C. T. Ratnam, A. L. Chuah, and W. Rashmi, "Waste tire rubber in polymer blends : A review on the evolution , properties and future," *Prog. Mater. Sci.*, vol. 72, pp. 100–140, 2015.

[35] K. Aoudia, S. Azem, N. Aït, M. Gratton, V. Pettarin, and S. Seghar, "Recycling of waste tire rubber : Microwave devulcanization and incorporation in a thermoset resin," *Waste Manag.*, 2016.

[36] J. E. Mark, B. Erman, C. M. Roland, and A. I. Isayev, "Chapter 15 – Recycling of Rubbers," in *The Science and Technology of Rubber*, 2013, pp. 697–764.

[37] A. M. Fernández, C. Barriocanal, and R. Alvarez, "Pyrolysis of a waste from the grinding of scrap tyres," *J. Hazard. Mater.*, vol. 203–204, pp. 236–243, 2012.

[38] F. A. López, T. A. Centeno, F. J. Alguacil, B. Lobato, and A. Urien, "Journal of Analytical and Applied Pyrolysis The GRAUTHERMIC-Tyres process for the

recycling of granulated scrap tyres," *J. Anal. Appl. Pyrolysis*, vol. 103, pp. 207–215, 2013.

[39] S. Ramarad, M. Khalid, C. T. Ratnam, A. L. Chuah, and W. Rashmi, "Waste tire rubber in polymer blends: A review on the evolution, properties and future," *Prog. Mater. Sci.*, vol. 72, pp. 100–140, 2015.

[40] P. J. H. van Beukering and M. A. Janssen, "Trade and recycling of used tyres in Western and Eastern Europe," *Resour. Conserv. Recycl.*, vol. 33, no. 4, pp. 235–265, Nov. 2001.

[41] W. J. Bodziak, "Tire tread and tire track evidence : recovery and forensic examination," p. 342, 2008.

[42] G. Ferrer, "The economics of tire remanufacturing," *Resour. Conserv. Recycl.*, vol. 19, no. 4, pp. 221–255, 1997.

[43] K. Fukumori, M. Matsushita, H. Okamoto, N. Sato, Y. Suzuki, and K. Takeuchi, "Recycling technology of tire rubber," *JSAE Rev.*, vol. 23, no. 2, pp. 259–264, 2002.

[44] Valorpneu, "Management of Used Tyres in Europe," 2017. [Online]. Available: http://www.valorpneu.pt/artigo.aspx?lang=ing&id_object=336&name=Management-of-Used-Tyres-in-Europe. [Accessed: 24-Mar-2017].

[45] A. I. Isayev, "15 – Recycling of Rubbers," in *Science and Technology of Rubber*, 2005, pp. 663–701.

[46] T. Amari, N. J. Themelis, and I. K. Wernick, "Resource recovery from used rubber tires," vol. 25, pp. 179–188, 1999.

[47] N. Sunthonpagasit and M. R. Duffey, "Scrap tires to crumb rubber: feasibility analysis for processing facilities," *Resour. Conserv. Recycl.*, vol. 40, no. 4, pp. 281–299, 2004.

[48] Northeast Recycling Council Inc, "Five Amazing Uses for Recycled Tires," 2015. [Online]. Available: https://nerc.org/news-and-updates/blog/nerc-blog/2015/11/20/five-amazing-uses-for-recycled-tires. [Accessed: 15-Apr-2017].

[49] K. Meardon, *Scrap Tire Technology and Markets*. Norwich, United States: William Andrew Publishing, 1993.

[50] A. C. Corredor-Bedoya, R. A. Zoppi, and A. L. Serpa, "Composites of scrap tire rubber particles and adhesive mortar – Noise insulation potential," *Cem. Concr.*

Compos., vol. 82, pp. 45–66, Sep. 2017.

[51] D. G. Pennington, R. C. Frazee, W. Chesbro, J. Gotch, P. Relis, and R. E. Chandler, “Effects of Waste Tires, Waste Tire Facilities, and Waste Tire Projects on the Environment,” no. May, pp. 1–52, 1996.

[52] M. A. Warith and S. M. Rao, “Predicting the compressibility behaviour of tire shred samples for landfill applications,” *Waste Manag.*, vol. 26, no. 3, pp. 268–276, Jan. 2006.

[53] K. Mogolodi Dimpe, A. Mpupa, and P. N. Nomngongo, “Microwave assisted solid phase extraction for separation preconcentration sulfamethoxazole in wastewater using tyre based activated carbon as solid phase material prior to spectrophotometric determination,” *Spectrochim. Acta - Part A Mol. Biomol. Spectrosc.*, vol. 188, pp. 341–348, Jan. 2018.

[54] L. Krayzelova, T. J. Lynn, Q. Banihani, J. Bartacek, P. Jenicek, and S. J. Ergas, “A Tire-Sulfur Hybrid Adsorption Denitrification (T-SHAD) process for decentralized wastewater treatment,” *Water Res.*, vol. 61, pp. 191–199, Sep. 2014.

[55] L. Chalker-Scott, “Rubber Mulch Use in Home Landscapes,” 2015.

[56] A. Guglielmotti, “Production of rubber parts by tyre recycling without using virgin materials,” *Plast. Rubber Compos.*, vol. 1, no. 41, p. 40:46, 2012.

[57] P. Lima, S. P. Magalhães Da Silva, J. Oliveira, and V. Costa, “Rheological properties of ground tyre rubber based thermoplastic elastomeric blends,” *Polym. Test.*, vol. 45, pp. 58–67, Aug. 2015.

[58] P. Lima, J. Oliveira, and V. Costa, “Partial replacement of EPDM by GTR in thermoplastic elastomers based on PP/EPDM: Effects on morphology and mechanical properties,” *J. Appl. Polym. Sci.*, 2014.

[59] P. S. Lima, J. M. Oliveira, and V. A. F. Costa, “Partial replacement of EPR by GTR in highly flowable PP/EPR blends: Effects on morphology and mechanical properties,” *J. Appl. Polym. Sci.*, 2015.

[60] R. Sonnier, E. Leroy, L. Clerc, A. Bergeret, and J. M. Lopez-Cuesta, “Polyethylene/ground tyre rubber blends: Influence of particle morphology and oxidation on mechanical properties,” *Polym. Test.*, vol. 26, no. 2, pp. 274–281, Apr. 2007.

[61] C. Xiaowei, H. Sheng, G. Xiaoyang, and D. Wenhui, “Crumb waste tire rubber surface modification by plasma polymerization of ethanol and its application on oil-well cement,” *Appl. Surf. Sci.*, vol. 409, pp. 325–342, Jul. 2017.

[62] L. W. McKeen and L. W. McKeen, “12 – Elastomers and Rubbers,” in *Permeability Properties of Plastics and Elastomers*, 2012, pp. 251–285.

[63] B. Abu-Jdayil, A. H. I. Mourad, and A. Hussain, “Investigation on the mechanical behavior of polyester-scrap tire composites,” *Constr. Build. Mater.*, vol. 127, pp. 896–903, 2016.

[64] J. Karger-Kocsis, L. M??sz??ros, and T. B??r??ny, *Ground tyre rubber (GTR) in thermoplastics, thermosets, and rubbers*, vol. 48, no. 1. 2013.

[65] M. Marvin, “Rubber Recycling,” *Rubber Chem. Technol.*, vol. 75, no. 3, 2002.

[66] S. Ramarad, M. Khalid, C. T. Ratnam, A. L. Chuah, and W. Rashmi, “Waste tire rubber in polymer blends: A review on the evolution, properties and future,” *Progress in Materials Science*, vol. 72. 2015.

[67] M. Razzaghi-Kashani, “Aramid-short-fiber reinforced Rubber as a tire tread composite,” *J. Appl. Polym. Sci.*, 2009.

[68] M. A. Martins, “Short sisal fiber-reinforced tire rubber composites: Dynamic and mechanical properties,” *Appl. Polym. Sci.*, vol. 91, no. 1, pp. 670–677, 2004.

[69] K. Aoudia, S. Azem, N. Aït Hocine, M. Gratton, V. Pettarin, and S. Seghar, “Recycling of waste tire rubber: Microwave devulcanization and incorporation in a thermoset resin,” *Waste Manag.*, 2016.

[70] V. L. Shulman, “Chapter 21 – Tyre Recycling,” in *Waste*, 2011, pp. 297–320.

[71] T. R. A. European, “Introduction to tyre recycling,” *ETRA News*. 2003.

[72] J. Scheirs, “Polymer Recycling: Science, Technology and Applications,” John Wiley., 1998.

[73] F. Quadrini, D. Gagliardi, G. M. Tedde, L. Santo, and E. Musacchi, “Direct molding of pavement tiles made of ground tire rubber,” in *AIP Conference Proceedings*, 2016, vol. 1769, no. 50002.

[74] J. Liu, “MANUFACTURING OF POLYPROPYLENE (PP)/ GROUND TIRE RUBBER (GTR) THERMOPLASTIC ELASTOMERS USING

ULTRASONICALLY AIDED EXTRUSION,” University of Akron , 2013.

[75] D. Mangaraj, “Role of Compatibilization in Recycling Rubber Waste by Blending with Plastics,” *Rubber Chem. Technol.*, vol. 78, no. 3, pp. 536–547, 2005.

[76] ASTM-E11-17, “Standard Specification for Woven Wire Test Sieve Cloth and Test Sieves,” *ASTM Stand. Guid.*, vol. E11-17, p. 9, 2013.

[77] Wikipedia, “Sieve Analysis,” 2017. [Online]. Available: https://en.wikipedia.org/wiki/Sieve_analysis. [Accessed: 28-Aug-2017].

[78] Scrap tire News, “The source for news and information about the scrap tire and rubber recycling industry,” 2016. [Online]. Available: http://www.scraptirenews.com/crumb.php. [Accessed: 02-Sep-2017].

[79] J. F. F. Horta, “Dissertação: Fabrico híbrido em grande escala de materiais compósitos,” IPleira, 2016.

[80] Coperion, “ZSK twin screw extruders.”

[81] Boymachines, “Dr. Boy Spritzgiessautomaten :: BOY 22,” *http://www.boymachines.com,* 2017. [Online]. Available: http://www.boymachines.com/products/boy-22-a/. [Accessed: 29-Mar-2017].

[82] O-Rings inc., “Rubber Hardness,” 2015. [Online]. Available: http://www.npl.co.uk/server.php?show=ConWebDoc.379. [Accessed: 26-Sep-2017].

[83] intertek.com, “Shore hardness ASTM d2240.” [Online]. Available: http://www.intertek.com/polymers/testlopedia/shore-hardness-astm-d2240/. [Accessed: 17-Jul-2017].

[84] G. Bracco and B. Holst, *Surface science techniques*, vol. 51, no. 1. 2013.

[85] Albright Silicone, “A guide to shore durometers.” [Online]. Available: https://albrightsilicone.com/a-guide-to-shore-durometers/. [Accessed: 18-Jul-2017].

[86] ASM International, “Introduction to Tensile Testing,” *Tensile Test.*, pp. 1–13, 2004.

[87] J. R. Davis, *Tensile Testing*. 2004.

[88] R. Gedney, “Measuring the plastic strain ratio of sheet metals.” pp. 3–5, 2006.

[89] Adana Science and Technology University, “True Stress and True Strain.”

[90] P. E. Labossiere, “Mechanical Properties and performance of materials,” 2007.

[91] Instron, “Modulus of Toughness,” 2017. [Online]. Available: http://www.instron.in/en-in/our-company/library/glossary/m/modulus-of-toughness. [Accessed: 01-Sep-2017].

[92] D. Roylance, “Stress-strain curves,” *Massachusetts Inst. Technol. study, Cambridge*, pp. 1–15, 2001.

[93] Bruker microCT, “Microtomography,” 2017. [Online]. Available: http://bruker-microct.com/company/methods.htm. [Accessed: 01-Sep-2017].

[94] A. Jurewicz, S. Jones, and A. Tsapin, “Locating Stardust-like particles in aerogel using x-ray techniques,” *Lunar Planet. Sci. XXXIV*, vol. XXXIV, no. 2003, pp. 2–3, 2003.

[95] R. Mizutani and Y. Suzuki, “X-ray microtomography in biology,” *Micron*, vol. 43, no. 2–3. pp. 104–115, Feb-2012.

[96] J. Bulantová, T. Macháček, L. Panská, F. Krejčí, J. Karch, N. Jährling, S. Saghafi, H. U. Dodt, and P. Horák, “Trichobilharzia regenti (Schistosomatidae): 3D imaging techniques in characterization of larval migration through the CNS of vertebrates,” *Micron*, vol. 83, pp. 62–71, Apr. 2016.

[97] J. C. Elliott and S. D. Dover, “X-ray microtomography,” *J. Microsc.*, vol. 126, no. 2, pp. 211–213, May 1982.

[98] SukanyaSatapathy, “Thermoplastic elastomers from waste polyethylene and reclaim rubber blends and their composites with fly ash,” *Process Saf. Environ. Prot.*, vol. 88, no. 2, pp. 131–141, Mar. 2010.

[99] Z. Hossain, B. Bairgi, and M. Belshe, “Investigation of moisture damage resistance of GTR-modified asphalt binder by static contact angle measurements,” *Constr. Build. Mater.*, vol. 95, pp. 45–53, Oct. 2015.

[100] S. M. Egodage, J. E. Harper, and S. Walpalage, “The development of rubber-thermoplastic blends from ground tyre rubber and waste polypropylene,” *J. Natl. Sci. Found. Sri Lanka*, vol. 37, no. 2, pp. 117–123, 2009.

[101] H. S. Liu, J. L. Mead, and R. G. Stacer, “Process Development of Scrap Rubber,” 2001.

[102] S. Ling Zhang, Z. Xiang Xin, Z. Xiu Zhang, and J. Kuk Kim, “Characterization of the

properties of thermoplastic elastomers containing waste rubber tire powder," *Waste Manag.*, vol. 29, pp. 1480–1485, 2009.

[103] GONCHARUK, "Effect of the specific area and the shape of rubber crumb on the mechanical properties of rubber-filled plastics," *Polym. Sci.*, vol. 40, no. 5–6, pp. 166–169, 1998.

[104] A. Puşcă, Ş. Bobancu, and A. Duţă, "MECHANICAL PROPERTIES OF RUBBER - AN OVERVIEW," *Bull. Transilv. Univ. Braşov*, vol. 3, pp. 52–2010.

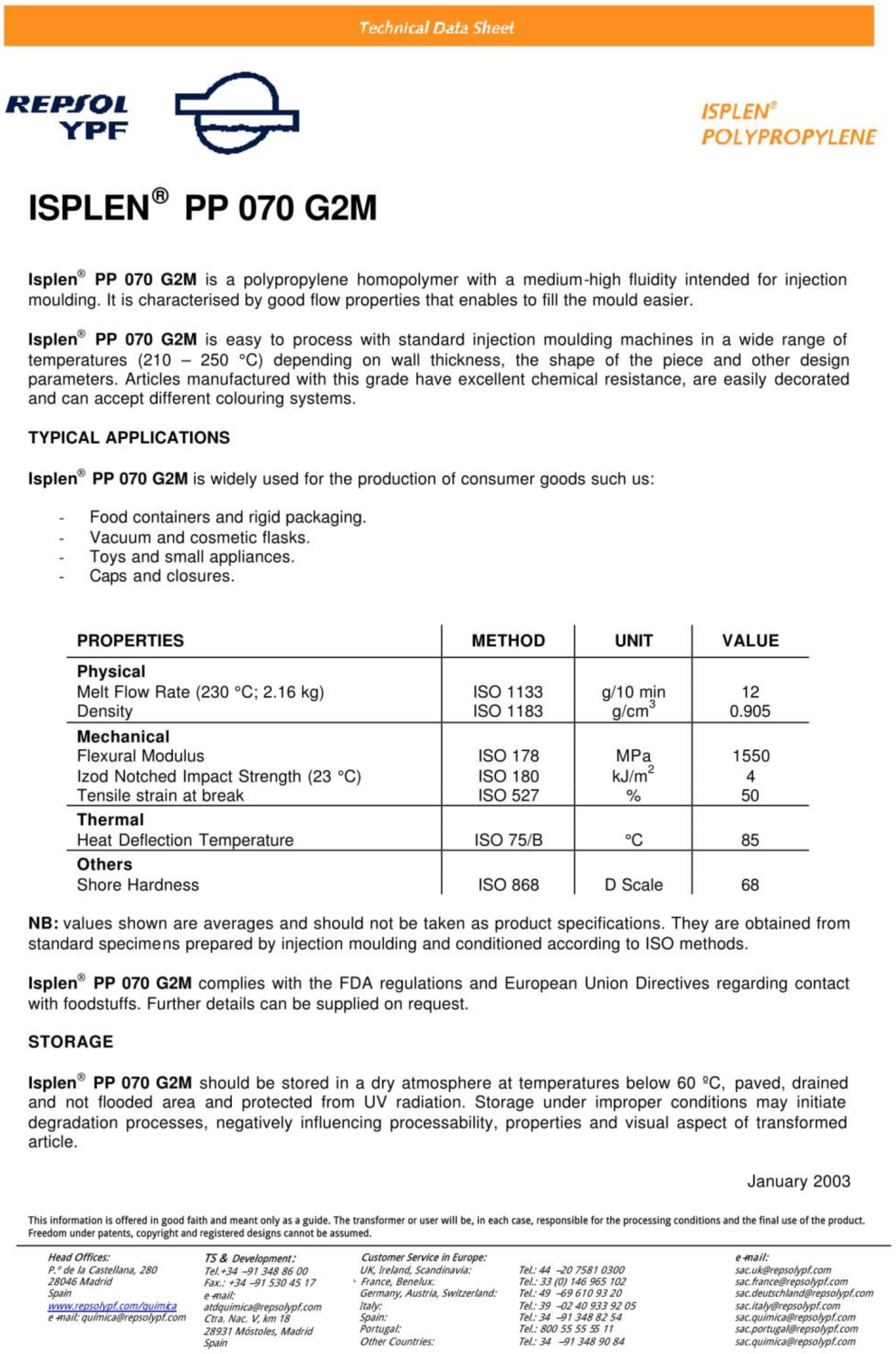
[105] C. A. Schneider, W. S. Rasband, and K. W. Eliceiri, "NIH Image to ImageJ: 25 years of image analysis," *Nat. Methods*, vol. 9, no. 7, pp. 671–675, Jul. 2012.

[106] B. Huang, X. Shu, and J. Cao, "A two-staged surface treatment to improve properties of rubber modified cement composites," *Constr. Build. Mater.*, vol. 40, pp. 270–274, Mar. 2013.

[107] I. Mangili, M. Lasagni, M. Anzano, E. Collina, V. Tatangelo, A. Franzetti, P. Caracino, and A. I. Isayev, "Mechanical and rheological properties of natural rubber compounds containing devulcanized ground tire rubber from several methods," *Polym. Degrad. Stab.*, vol. 121, pp. 369–377, 2015.

Appendices

Appendix A



Technical Data Sheet

REPSOL YPF

ISPLEN®
POLYPROPYLENE

ISPLEN® PP 070 G2M

Isplen® PP 070 G2M is a polypropylene homopolymer with a medium-high fluidity intended for injection moulding. It is characterised by good flow properties that enables to fill the mould easier.

Isplen® PP 070 G2M is easy to process with standard injection moulding machines in a wide range of temperatures (210 – 250 °C) depending on wall thickness, the shape of the piece and other design parameters. Articles manufactured with this grade have excellent chemical resistance, are easily decorated and can accept different colouring systems.

TYPICAL APPLICATIONS

Isplen® PP 070 G2M is widely used for the production of consumer goods such us:

- Food containers and rigid packaging.
- Vacuum and cosmetic flasks.
- Toys and small appliances.
- Caps and closures.

PROPERTIES	METHOD	UNIT	VALUE
Physical			
Melt Flow Rate (230 °C; 2.16 kg)	ISO 1133	g/10 min	12
Density	ISO 1183	g/cm^3	0.905
Mechanical			
Flexural Modulus	ISO 178	MPa	1550
Izod Notched Impact Strength (23 °C)	ISO 180	kJ/m^2	4
Tensile strain at break	ISO 527	%	50
Thermal			
Heat Deflection Temperature	ISO 75/B	°C	85
Others			
Shore Hardness	ISO 868	D Scale	68

NB: values shown are averages and should not be taken as product specifications. They are obtained from standard specimens prepared by injection moulding and conditioned according to ISO methods.

Isplen® PP 070 G2M complies with the FDA regulations and European Union Directives regarding contact with foodstuffs. Further details can be supplied on request.

STORAGE

Isplen® PP 070 G2M should be stored in a dry atmosphere at temperatures below 60 ºC, paved, drained and not flooded area and protected from UV radiation. Storage under improper conditions may initiate degradation processes, negatively influencing processability, properties and visual aspect of transformed article.

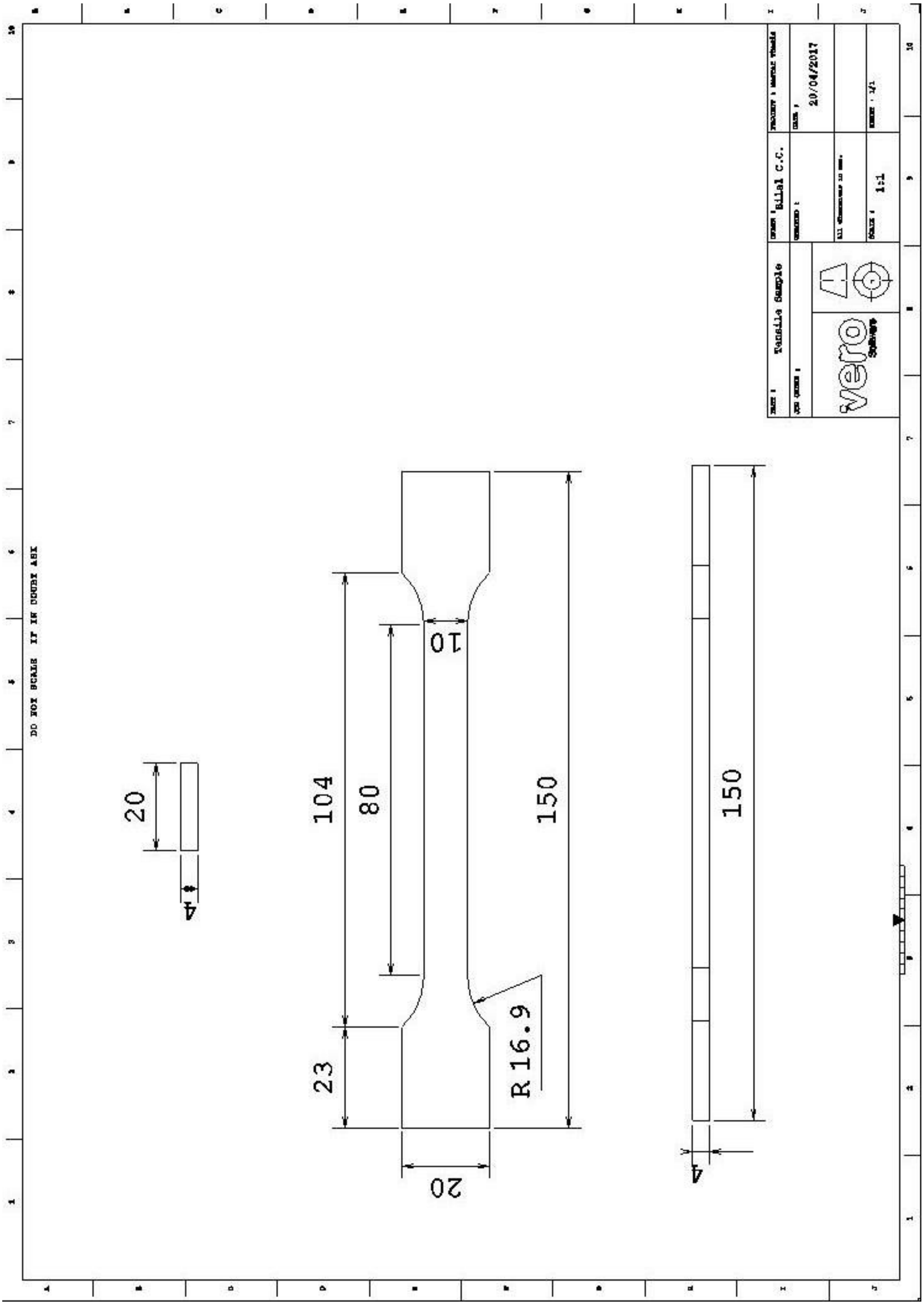
January 2003

Head Offices:
P.º de la Castellana, 280
28046 Madrid
Spain
www.repsolypf.com/quimica
e-mail: quimica@repsolypf.com

TS & Development:
Tel.+34 –91 348 86 00
Fax.: +34 –91 530 45 17
e-mail:
atdquimica@repsolypf.com
Ctra. Nac. V, km 18
28931 Móstoles, Madrid
Spain

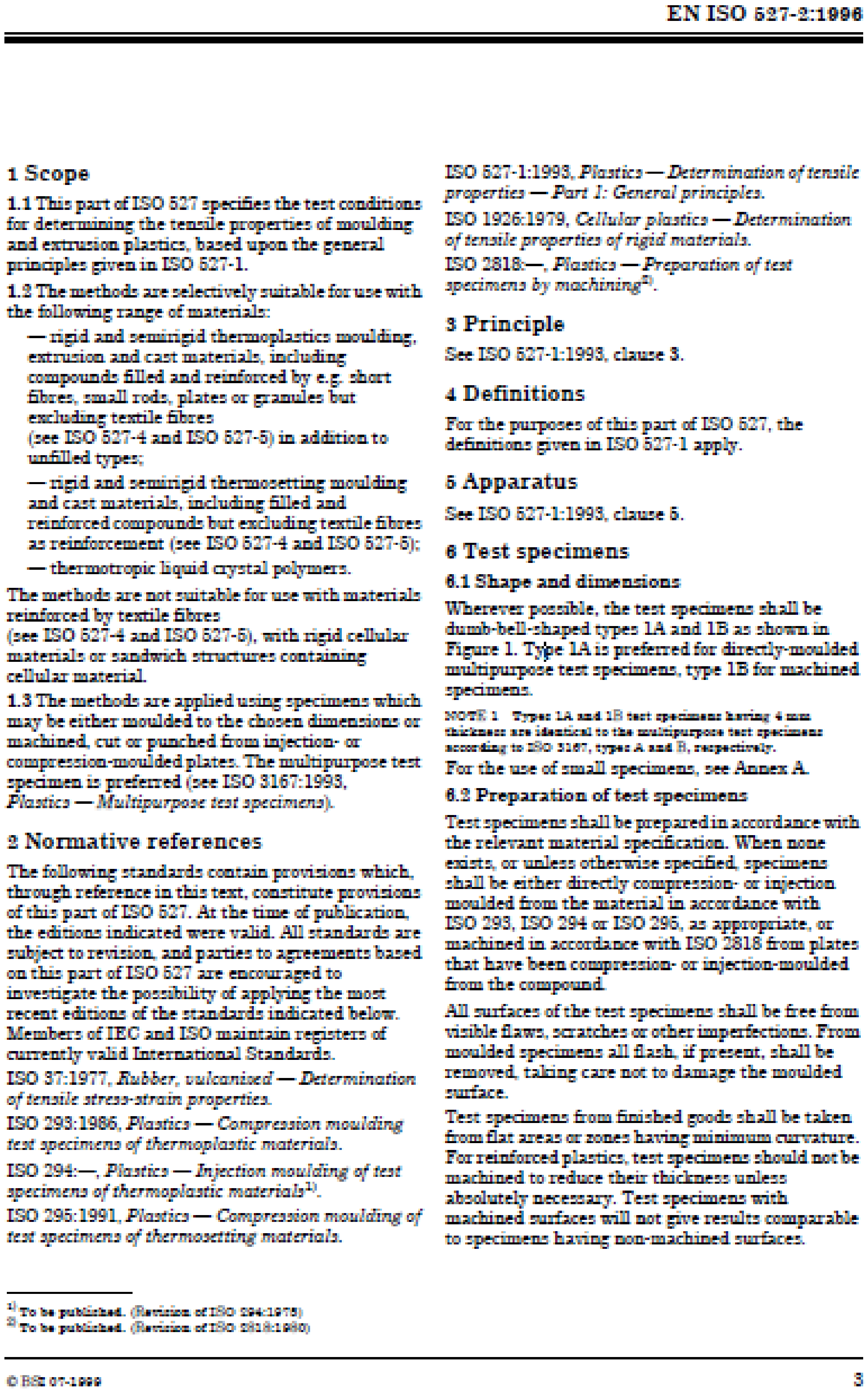
Customer Service in Europe:		*e-mail:*
UK, Ireland, Scandinavia:	*Tel.: 44 –20 7581 0300*	*sac.uk@repsolypf.com*
France, Benelux:	*Tel.: 33 (0) 146 965 102*	*sac.france@repsolypf.com*
Germany, Austria, Switzerland:	*Tel.: 49 –69 610 93 20*	*sac.deutschland@repsolypf.com*
Italy:	*Tel.: 39 –02 40 933 92 05*	*sac.italy@repsolypf.com*
Spain:	*Tel.: 34 –91 348 82 54*	*sac.quimica@repsolypf.com*
Portugal:	*Tel.: 800 55 55 55 11*	*sac.portugal@repsolypf.com*
Other Countries:	*Tel.: 34 –91 348 90 84*	*sac.quimica@repsolypf.com*

Appendix B



DO NOT SCALE IF IN DOUBT ASK
20
4
104
80
150
23
R 16.9
10
20
150
4
Tensile Sample
Bilal C.C.
20/04/2017
1:1
1/1
vero Software

Appendix C



1 Scope

1.1 This part of ISO 527 specifies the test conditions for determining the tensile properties of moulding and extrusion plastics, based upon the general principles given in ISO 527-1.

1.2 The methods are selectively suitable for use with the following range of materials:

- rigid and semirigid thermoplastics moulding, extrusion and cast materials, including compounds filled and reinforced by e.g. short fibres, small rods, plates or granules but excluding textile fibres (see ISO 527-4 and ISO 527-5) in addition to unfilled types;
- rigid and semirigid thermosetting moulding and cast materials, including filled and reinforced compounds but excluding textile fibres as reinforcement (see ISO 527-4 and ISO 527-5);
- thermotropic liquid crystal polymers.

The methods are not suitable for use with materials reinforced by textile fibres (see ISO 527-4 and ISO 527-5), with rigid cellular materials or sandwich structures containing cellular material.

1.3 The methods are applied using specimens which may be either moulded to the chosen dimensions or machined, cut or punched from injection- or compression-moulded plates. The multipurpose test specimen is preferred (see ISO 3167:1993, *Plastics — Multipurpose test specimens*).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 527. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 527 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 37:1977, *Rubber, vulcanized — Determination of tensile stress-strain properties.*

ISO 293:1986, *Plastics — Compression moulding test specimens of thermoplastic materials.*

ISO 294:—, *Plastics — Injection moulding of test specimens of thermoplastic materials*[1].

ISO 295:1991, *Plastics — Compression moulding of test specimens of thermosetting materials.*

ISO 527-1:1993, *Plastics — Determination of tensile properties — Part 1: General principles.*

ISO 1926:1979, *Cellular plastics — Determination of tensile properties of rigid materials.*

ISO 2818:—, *Plastics — Preparation of test specimens by machining*[2].

3 Principle

See ISO 527-1:1993, clause 3.

4 Definitions

For the purposes of this part of ISO 527, the definitions given in ISO 527-1 apply.

5 Apparatus

See ISO 527-1:1993, clause 5.

6 Test specimens

6.1 Shape and dimensions

Wherever possible, the test specimens shall be dumb-bell-shaped types 1A and 1B as shown in Figure 1. Type 1A is preferred for directly-moulded multipurpose test specimens, type 1B for machined specimens.

NOTE 1 Types 1A and 1B test specimens having 4 mm thickness are identical to the multipurpose test specimens according to ISO 3167, types A and B, respectively.

For the use of small specimens, see Annex A.

6.2 Preparation of test specimens

Test specimens shall be prepared in accordance with the relevant material specification. When none exists, or unless otherwise specified, specimens shall be either directly compression- or injection moulded from the material in accordance with ISO 293, ISO 294 or ISO 295, as appropriate, or machined in accordance with ISO 2818 from plates that have been compression- or injection-moulded from the compound.

All surfaces of the test specimens shall be free from visible flaws, scratches or other imperfections. From moulded specimens all flash, if present, shall be removed, taking care not to damage the moulded surface.

Test specimens from finished goods shall be taken from flat areas or zones having minimum curvature. For reinforced plastics, test specimens should not be machined to reduce their thickness unless absolutely necessary. Test specimens with machined surfaces will not give results comparable to specimens having non-machined surfaces.

---

[1] To be published. (Revision of ISO 294:1975)

[2] To be published. (Revision of ISO 2818:1980)

6.3 Gauge marks

See ISO 527-1:1993, subclause 6.3.

6.4 Checking the test specimens

See ISO 527-1:1993, subclause 6.4.

7 Number of test specimens

See ISO 527-1:1993, clause 7.

8 Conditioning

See ISO 527-1:1993, clause 8.

9 Procedure

See ISO 527-1:1993, clause 9.

For the measurement of the modulus of elasticity, the speed of testing shall be 1 mm/min for specimen types 1A and 1B (see Figure 1). For small specimens see Annex A.

10 Calculation and expression of results

See ISO 527-1:1993, clause 10.

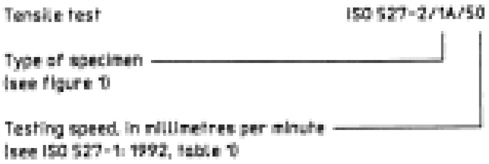
11 Precision

The precision of this test method is not known, because interlaboratory data are not available. When interlaboratory data are obtained, a precision statement will be added with the next revision.

12 Test report

The test report shall include the following information:

a) a reference to this part of ISO 527, including the type of specimen and the testing speed according to:



For items b) to q) in the test report, see ISO 527-1:1993, 12 b) to q).