Tampere University

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PUNCTURE RESISTANCE OF PAPERS USED IN PACKAGING

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ABSTRACT

Lucas do Carmo Tinoco: Puncture resistance of papers used in packaging
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Puncture resistance (PR) is a key material property in order to evaluate how a component can withstand external or internal piercing loads without being damaged by those. A package material must have high PR to be effective as a packaging.

This thesis focused on the puncture resistance of papers used in packaging. It had different objectives: to design and manufacture the testing equipment; to analyze how different parameters affect the puncture resistance; to determine which mechanical properties correlates better with PR; and to evaluate possible causes for the differences in PR of the papers tested.

First, all the tool necessary for the testing were manufactured – probe holder, sample holder and penetration probes. These probes were produced in order to emulate different damages a package can suffer in real conditions.

In total, it was used five different probes: one flat probe; and four hemispherical probes. They all had the same base dimensions, but their tip radius varied. Only one probe had a soft tip -to emulate the impact of a chocolate bar into its package-, the others were hard tip probes.

It was determined that stress concentration is the main factor which justifies the difference in a paper puncture resistance when using different probes. A probe that causes higher stress concentration to a paper will pierce it easier.

The flat probe caused less stress concentration than the hemispherical ones, thus provided higher PR. Also, it was evaluated that probes with the same tip geometry will affect a paper' puncture resistance in a direct proportion to its tip dimension. Finally, it was concluded that the tip's material does not have more influence than its shape.

The tensile properties of the samples were calculated to check their correlation with PR. Tensile Energy Absorption (TEA) proved to be the property with higher correlation. This correlation was used to determinate which are the driving factors that justifies the PR differences between the tested papers, taking in consideration that the driving factor for TEA would be similar to PR. Those are the pulp used, the quantity of filler added, and the refining process. In order to produce a paper with high puncture resistance it is necessary to reduce the amount of filler and to wisely select the pulp and its refining process.

The papers selected for this thesis had different characteristics, such as grammage and presence of coating/glazed side in order to analyze if those effect its puncture resistance value. It was analyzed that a particular paper will have its puncture resistance directly proportional to its grammage. Also, for the papers tested the presence of coating/glazed side did not show influence on PR.

Furthermore, different papers' types were used to check the influence of moisture content in PR. It was concluded that higher moisture content increases a paper's puncture resistance, but this relation is not directly proportional. Finally, it was concluded that in overall unbleached paper tends to provide higher PR than bleached paper, and that the material used (pulp), or even a combination of it, will influence on the paper's puncture resistance.

Keywords: Packaging, paper, puncture resistance, stress concentration, tensile energy absorption

PREFACE

This thesis was a collaboration project between Tampere University, Nestlé, and Mondi; thus, I would like to thank them for this opportunity.

Several people have helped me during this project work, so I would like to thank TAU staff - Kati Mökkönen, Säde Mäki, Rauli Mäkinen - to be very supportive and helpful during this period.

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Tampere, 9 November 2020

Lucas do Carmo Tinoco

CONTENTS

LIST OF SYMBOLS AND ABBREVIATIONS

Adv.	Advantaged
CD	Cross Direction
EN	European Standard
HT	Heat Sealable
ISO	International Organization for Standardization
MD	Machine Direction
MF	Machine Finished
MG	Machine Glazed
ML	Maximum Load
MS	Maximum Strain
PR	Puncture Resistance
RBA	Relative Bonded Area
RH	Relative Humidity
SBS	Solid Bleached Board
SC	Super Calendered
TEA	Tensile Energy Absorption
UV	Ultraviolet

δ'_T	Mean Elongation at Break
ε_T	Strain at Break
σ_T^b	Tensile Strength
σ_T^W	Tensile Index
b	Initial test piece width
F'_T	Mean Maximum Tensile Force
l	Initial test piece length
U'_T	Mean area below the elongation-force curve
w	Grammage
W_T^b	Tensile Energy Absorption
W_T^W	Tensile Energy Absorption Index
P_{CD}	Mean values of Cross Directions readings
$Pgeo$	Geometric mean
P_{MD}	Mean values of Machine Directions readings

1. INTRODUCTION

Consumerism is known to increase yearly. One of the consequences of this buyer behaviour is the expansion of waste produced, such as packaging materials. Packages can be made of different materials, the ones which are not sustainable are problematic, thus more environmentally friendly options are being requested by the society.

Plastics packages can intensify environmental problems such as the plastic pollution if these are not recycled or made of biodegradable plastic. Plastic pollution is the accumulation of plastic and plastic products in the ecosystem which leads to negative consequences to nature, wildlife, and humans [1].

It is important to use options that are greener, to do so, it is necessary to reduce the amount of plastic in packaging, using only when it is needed for its properties. One viable solution is to use fiber-based materials such as paper and paperboard. They are renewable, reusable, and recyclable [2].

Packages are important to guarantee the quality of the product and to facilitate the transport and handling. Packaging made of fiber-based materials must be effective in all of these requirements, thus it is necessary to manufacture them adequately.

Packages can suffer damages during transportation, handling and even from the material that is packed inside. Any damage that occur to them will compromise the product's quality and its safety. It is essential that the packaging used has high enough strength to avoid damages. One way to analyse paper's strength, which is used as packaging, is to measure its puncture resistance.

Puncture resistance is the ability of a material to resist the penetration from an object. In this thesis, different papers will be tested to analyse their puncture resistance, thus their suitability as packaging material. Furthermore, it will be studied how different properties, such as probe design and moisture content, influence on puncture resistance values.

2. PAPER MATERIAL

2.1 Basics of paper manufacturing & packaging paper

Paper and paperboard manufacturing use different types of components to product a myriad of paper types. Pulp and paper are produced using materials that contains cellulose fibers, mainly wood and recycled paper. Pulp and paper production are basically a two-stage process, pulping and papermaking [3].

The pulping stage aims at separating fibers from the wood matrix, in a manner that the fibers' integrity is suitable for the papermaking process [4]. This process can be done mechanically or chemically, known as mechanical pulping and chemical pulping respectively [5]. The pulping method selection is done according to the used raw material and the wanted final product [6].

Mechanical pulping is a process in which logs or chips are ground or grated. This leads to a high yield pulp, approximately 95% [5]. Pulp produced by this process presents low quality and strength, being mainly used in newsprints [6].

Chemical pulping consists of blending the raw material with cooking chemical in controlled pressure and temperature conditions to produce several particular pulps. In this, the used chemicals will cause lignin degradation or dissolving that separates the woods fibers. [5,6]. Pulp yield is between 45% and 55% according to the desired delignification [5].

After the pulping, the bleaching process is performed. Bleaching is a process by which whiteness is increased by either modifying or removal of the colored molecules that are formed in the cooking [5]. This operation is important because it creates papers that are whiter, softer, brighter than when bleaching is not done [6].

Each bleaching process is done according to the pulping method used. Mechanical pulps bleaching is generally done by oxidation or reduction of pulp chromophores, this process only discolors the lignin, not removing them. On the other hand, in the chemical pulp bleaching process, residual lignin is eliminated [5].

The next stage is the papermaking process, which is further divided into four steps: preparation of the stock; formation of sheets; pressing and drying; sizing and coating [5].

Stock preparation is the place where pulp is prepared to be used in paper machines. This preparation involves the mixture of different pulps, chemicals additions and dilution. The quality of the stock (pulp) determines the properties of paper [4].

Formation of sheets is dependent on the water removal ability and the wire surface pulp repartition. The headbox, which is a paper machine component, permits the pulp deflocculating and the pulp spreading out at the wire surface. This component strongly controls the paper sheet basic weight. Afterwards, the pulp deposition on the wire, a large water quantity needs to be removed [5].

Pressing is a process by which water is removed from the paper web. The paper web moves between two rolls that are pressed together. After this stage, the paper web humidity is around 35-45%. Later, the drying section removes more water until its humidity reaches 4%. The drying process is done by heating of the paper web [5].

Finally, different processes can be applied. Sizing provides water resistance for the paper surface, which is needed for paper used for printing or writing [4]. Also, calendering can take place, which turns the paper's surface smoother, thus increasing its printability and gloss [7]. Coating can be applied for several reasons, such as increasing printability and resistance to water and grease [8].

Paper can be coated by polymer or pigments. Pigment coating is usually used to improve a paper's functionality, printability or even its visual properties [9]. On the other hand, Polymer coating improves its barrier properties (grease, water, oxygen, etc.), and can be applied by different methods such as dispersion coating, extrusion coating and lamination [10].

Dispersion coating is the application of a latex coating on a paper or paperboard surface. This process aims at the creation of barrier layers that can protect against grease, water, gas, etc. [11]. It is commonly used for sacks, many kinds of wrappings, disposables, candy boxes, bakery products, packages for greasy food [10].

Extrusion coating is a process by which a polymer is extruded as a film coating that is applied onto a substrate (paper or paperboard). The polymer used should have both high melt temperature and high molecular weight. This method is used for different applications, for example in food industry it can be used for coating the paperboard used in juice and milk cartons [10,12].

Lamination is a process of adhering a plastic film into paper or paperboard by application of heat and pressure. Laminated paper has protection against moisture scratch, tear, wear. Furthermore, lamination improves the colors and appearance of the printed paper [13]. Lamination is used to manufacture laminates packaging as seen on Figure 1 [14].

Figure 1. *Packaging laminates from Mondi [14].*

Different fiber-based substrates are suitable for lamination. The most common materials are Machine Glazed (MG) paper, Machine Finished (MF) paper, Kraft paper and Solid bleached board (SBS) paperboard [10]. Several flexible packages and liquid packages are manufactured with paper or paperboard laminates [10,13].

Paper and paperboard are manufactured using the same technology methods. There are several types of paper and paperboard to meet the markets of different requirements. The election is based on the fiber selection, additives and chemicals needed, different coatings, and with different mechanical surface finish, such as Super Calendered (SC), MG and MF [10].

Fiber-based packaging material is usually used in many different industries – food, chemicals, construction. This research project focuses on the food industry, more specifically on packaging paper.

Packaging papers require high strength, in order to have these characteristic they can be produced by using virgin pulps, kraft, recycled fibers, or even from a mixture of recycled fibers and chemical pulp. Normally mechanical pulps are not used for this paper type [10].

Properties such as tear strength, bursting strength and puncture resistance are important elements in packaging papers. Other properties, such as water impermeability, and water repellency can be obtained by specific coating, or by the addition of special additives into the pulp. Examples of packaging papers are Kraft paper, bleached paper, coated paper, and glassine paper [10].

2.2 Fiber network and physical structure of paper

Physical properties of the finished paper are mainly influenced by the fiber properties and its structure (fiber distribution in the sheet). This is true while not taking to consideration the influence of filler additives and other elements' effects [15].

Paper is a network of fibers that are arranged in a random distribution. Paper network is planar and essentially two-dimensional (Figure 2), considering that fibers have greatly higher thickness compared to the paper sheet thickness. Several paper properties are dependent of this two-dimensional structure, but the three-dimension porous structure is significant as well. These pores are responsible for paper's bulky, rigid, and opaque structure [16].

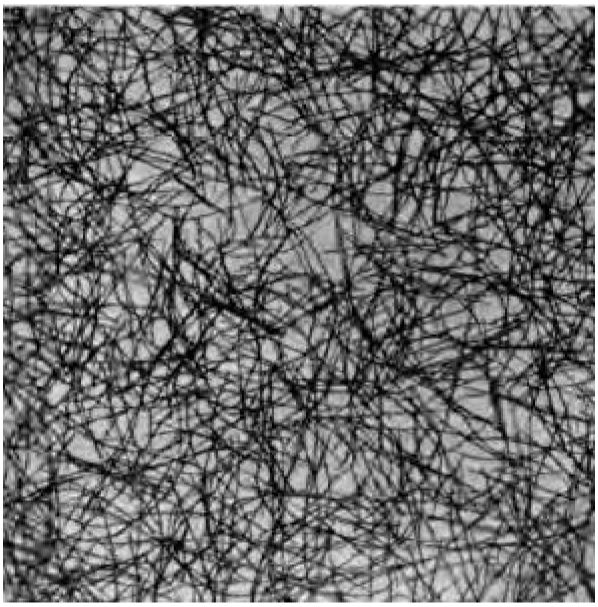


Figure 2. *2D fiber network [16].*

It is necessary to analyse the geometric characteristics of the fiber network. On the two-dimensional structure level, fibers are considered as straight-line sections of constant length. It is noteworthy that for this geometry neither distribution nor different fiber properties are relevant. Oppositely, on the three-dimensional structure, the fiber conformability and thickness are essential [16].

It is worthy to mention that paper structure is not fully random. The spatial basis weight distribution of paper, known as formation, does not follow a complete random distribution. During the paper production fibers are gathered in flocs of particular size. Both fiber flocculation tendency and web forming process hydrodynamics influence on the paper's local basis weight. It is more likely to find, at short distance, alike basis weights number [16].

Mechanical properties of paper are dependent on the bonding degree (or connectivity) of the fiber network. A fair amount of bonds between the fibers are needed for the network to have cohesion [16].

The bonding degree of a paper can be measured by the relative bonded area (RBA). RBA is, by definition, the amount of fiber bonded surface area divided by the fiber's total surface area. In the two-dimensional structure, once there is not space between fibers in the thickness direction, bonds are formed in all of the fiber crossing [16].

The three-dimensional network paper structure rules both optical properties and density directly and both dimensional stability and mechanical properties indirectly through the relative bonded area [16].

2.3 Paper properties and factors affecting them

This chapter deals with several paper properties and how these can be affected by distinct factors. Furthermore, it illustrates how these properties influence on paper strength.

2.3.1 Fiber orientation

Fiber orientation is an important feature for understanding paper structure and its effects in paper's properties. Paper produced by machines have an anisotropic structure – higher quantity of fibers is aligned near the Machine Direction (MD) compared with the Cross Direction (CD) [16], as illustrated in Figure 3.

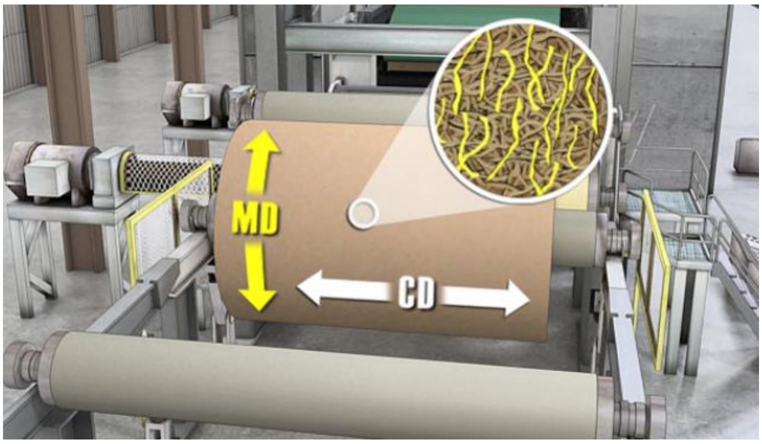


Figure 3*. Machine-direction and Cross-machine direction [17].*

Fiber orientation has a large impact on physical properties such as drying shrinkage, wet straining and anisotropy in strength [18]. It can occur by different factors, but the most important is the difference in speed between wire and suspension jet [19].

Mechanical properties and paper dimensional stability are directly influenced by the orientation of fibers. Figure 4 illustrates a typical stress-strain curve for paper in Machine and Cross direction. In this, it is possible to see the difference in Tensile Strengths between directions, which is higher in the Machine Direction [20].

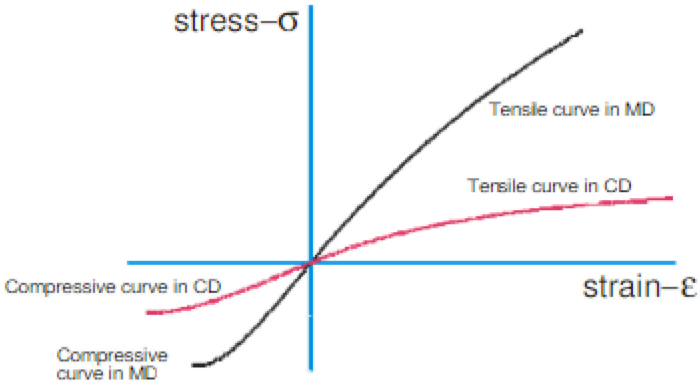


Figure 4. *Typical paper's Strain vs. Stress Curve [20].*

2.3.2 Fiber strength

Fiber properties have an influence on paper fracture properties. In overall, the decrease of fiber strength and length results in reduction of fracture energy. Also, the increase of fiber curls leads to higher breaking strain, higher fracture energy and lower breaking tension of paper network [21].

Any fiber defect decreases fiber strength intensively. Not only the visible defects, but also the defects caused internally in the fiber structure. Chemical pulping damages the fiber structure, thus reduces its strength. Furthermore, during this pulping method, fiber deformations (such as kinks and curl) happen which affects the fiber networks [21].

It is worthy to mention that pulp fibers which are industrially made suffer mechanical damage in the fiber line. Those damages weaken the fibers [21].

2.3.3 Formation

Paper sheets are made of fibers, fiber fragments, chemical additives, and mineral fillers. During web formation all these elements are settled down randomly on the web, which leads to a nonuniform particle distribution. Paper formation is this particular distribution that causes variability of the basis weight. This process is influenced by fiber flocculation [16].

Paper formation is influenced by two different factors. The first one is the quality of the wood pulp and all the elements added in the paper production. The other is the moving wire's speed [22].

Figure 5 exposes paper sheets with good and poor formations. The paper with good formation presents fiber's uniform alignment, on the other hand, the one with poor formation have uneven and blotchy fibers [22].

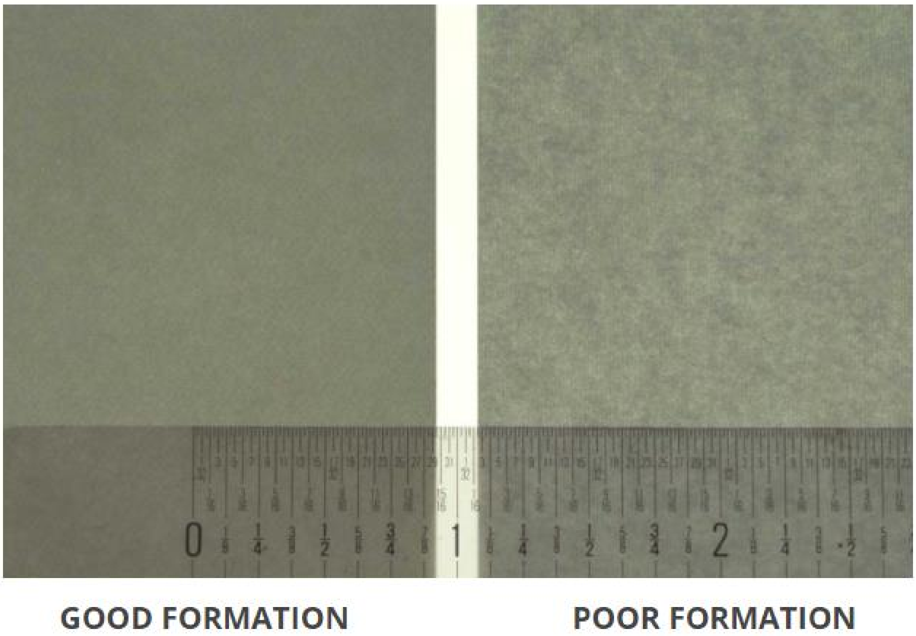


Figure 5. *Paper sheets with good and bad formation [22].*

Many paper properties are affected by paper formation, for example tearing resistance and tensile strength. If the formation becomes more nonuniform, then the tensile strength decreases [16,23]. Furthermore, a good formation will improve paper print results, paper smoothness and paper gloss [24].

2.3.4 Hardwood vs. Softwood

Wood can be divided in two types: Softwood and Hardwood. The selection of the wood type used in papermaking will influence on the paper physical properties. Moreover, each kind can be used to manufacture different paper varieties [19].

Softwood are originating in gymnosperms trees. It has simple wood anatomy, which consists mainly of longitudinal fibers tracheids (2,5 - 7mm long), and fewer amount of ray cells and resin cells [25].

Hardwood are originally from angiosperms trees. This type has a complex wood anatomy, consisting of fiber cells, vessels elements, ray cells and parenchyma cells. Hardwoods fibers vary form 0,9 – 1,5mm long, which produces a smoother paper, but with lower strength compared to softwood fibers [25].

Paper made of softwood has high folding strength, good tensile strength, folding strength, and printability. Paper from hardwood is loose with high thickness, stiffness and opaqueness, and strong absorptivity [26].

Softwood pulp is the best type to produce paper, which is used for different types, such as coated paper, text paper, offset paper, etc. Hardwood pulp produces paper with lower quality, but still with good characteristics, such as printing paper [26,27].

Softwood and hardwood fibers can be mixed to achieve a paper with specific strength, writing surface, etc. [27]. Figure 6 depicts how this fiber combination affects some properties. Paper with higher softwood content will be stronger and brighter, on other hand, papers with high amount of hardwood has better formation and it is not as strong [28].

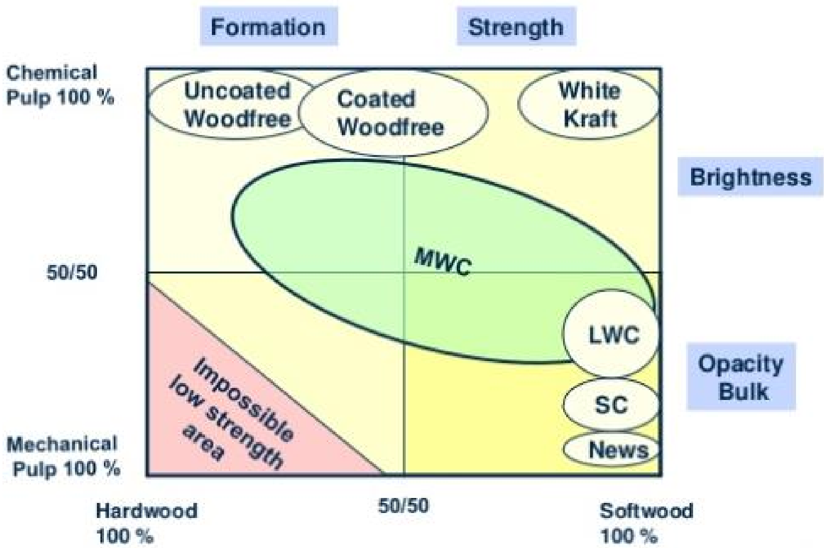


***Figure 6**. Fiber combination used in white paper from Europe [28].*

2.3.5 Bleached vs. Unbleached paper

Pulp bleaching is a process in papermarking that lighten the pulped wood particles. This is an important stage when both white paper and colored paper are wanted, considering that to dye a paper, it is required to have a bleached surface. Furthermore, pulp bleaching removes impurities and helps achieving the paper product's chemical balance [29].

Bleached paper is made using chemical pulps which are soft, white, and capable of receiving special chemical that can provide particular functional properties. This paper type is used in packaging which the requirements are for good printing and some functional property. These are commonly used in flexible packages, pouches, labels, cosmetics, laminates [10].

Unbleached paper is commonly stiffer, stronger and more course compared to bleached paper, but has fewer applications than bleached one [30]. However, unbleached paper is a more environmentally friendly option compared to bleached paper [31].

Figure 7 illustrates that paper made from bleached chemical pulps can be weaker than unbleached chemical paper. Also, it can also be observed the high difference in whiteness between them [32].

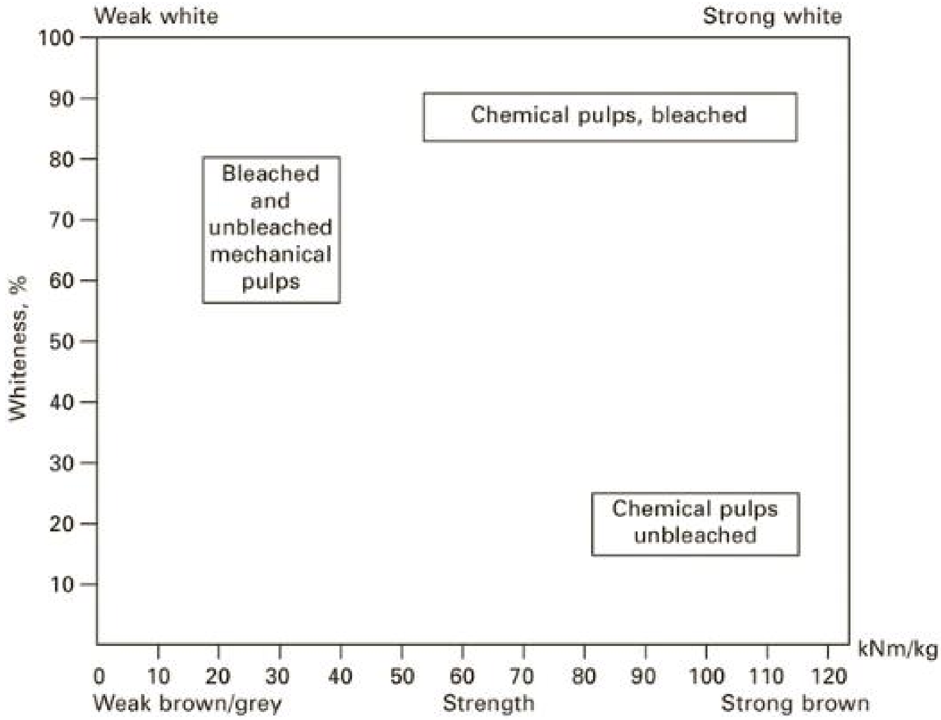


Figure 7. *Scheme comparation of strength and whiteness for different pulps [32].*

2.3.6 Papermaking additives (fillers and chemicals)

Nowadays papermaking relies on the presence of additives. The additives are fillers and chemicals, being the former presented in higher concentration. The introduction of additives to papers has economic reasons and it also improves the quality of the final product [33].

Fillers are common in paper composition. Fillers are white, fine pigment powders which can be manufactured from natural mineral or from different raw materials synthetically. The reasons for using them are various: reduction of cost in production; improvement of different properties (printability, gloss, brightness, evenness of formation, etc.); filing of space between fibers [33].

However, fillers have also negative effects on paper and its process. Strength properties are lowered with the addition of fillers into the paper structure, once fillers are incapable of forming considerable bond to fibers. The addition of filler decreases the amount of fibers in the network per unit volume, which reduces strength properties [33].

The magnitude of the loss is different for each strength property. Figure 8 illustrates how different strength properties are influenced by the filler amount. Properties such as TEA Index and Burst Index decreases strongly with higher filler content [33].

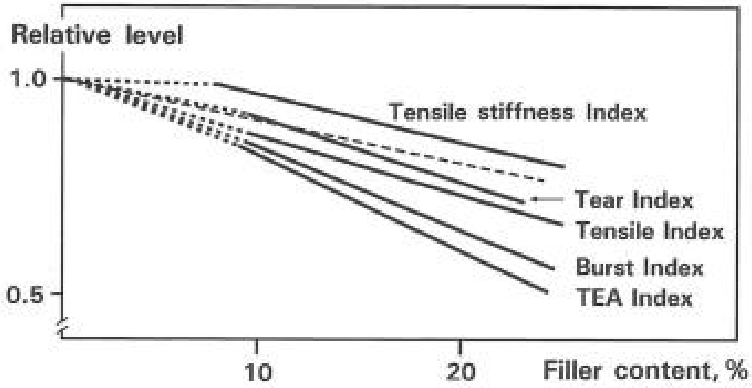


Figure 8. *How different strength properties are affected by filler loading [33].*

Papermaking chemicals can be divided into two groups: functional chemicals and process chemicals. Functional chemicals can improve paper's strength, hydrophobicity and optical properties. On the other hand, process chemicals improve the papermaking process. It is noteworthy that this division is not very strict because one chemical can improve both functional and process effects, as seen on Table 1 [33].

Table 1. *Functional effects and process effects of chemicals used in papermaking [33].*

Chemical	Functional effect	Process effect
Dry-strength	++	+
Wet-strength	+++	+
Sizing (hydrophobation)	+++	+
Dyes and optical brighteners (OBAs)	++	
Coating color additives	+++	++
Fixatives		+++
Retention, drainage, and formation aids (RDFs)	+	+++
Defoaming agents		+++
Biocides		+++
Dispersing and detackifying agents	+	++
Detergents		+++

2.3.7 Pulp refining

Pulp refining is an important mechanical treatment which improves fibers strength, thus raise the quality of the paper produced. This treatment increases fiber-to-fiber bonds strength due to the increase of the fiber surface area, which makes fibers more flexible to accommodate with each other. This phenomenon results in a denser sheet and improvement in the bonding surface area [25].

The refining process, due to the increase of the fiber bonding, results in increase of folding, burst tensile endurance, and in tensile energy absorption. However, the tearing resistance also reduces, caused by the individual fiber's attrition in strength [25].

2.3.8 Drying

The drying process has a crucial importance on the paper quality. During this stage fiber shrinks because of the water evaporation which causes stress on the web. It is possible to improve the strength properties in the network by controlling the amount of stress during the drying stage [34].

The fiber shrinkage during drying should be reduced, if so, the fibers network will have higher tensile strength, higher modulus of elasticity and improved dimensional stability. Different paper properties are also caused by diverse drying stresses during this process [34].

2.3.9 Moisture Content

Moisture content is the amount of water present in the paper, and it can vary according to the papermaking process [35]. Fiber-based package materials quickly absorbs moisture because its cellulose fibers are very porous. The absorption can happen when paper is in contact with food that has high moisture content, or by absorbing moisture from an environment with high humidity [36].

In most cases, different mechanical properties are strongly affected by the moisture content such as, tensile properties, dimensional stability, and compression strength [36]. Moisture contest is dependent of its environment relative humidity.

Figure 9 illustrates how different paper properties behaviour under different relative humidity value.

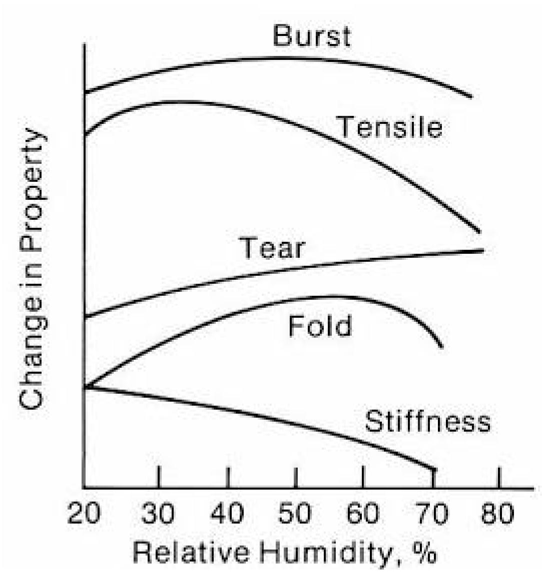


Figure 9. *Paper's changes in properties according to relative humidity amount [37].*

Burst Strength is a function of both stretch and tensile strength. Burst increases until tensile and/or stretch is increasing. Tensile start to decrease at about 35% Relative Humidity (RH), but stretch is still increasing, thus burst increases as well. After 55% RH, burst starts to decrease, even though stretch is still increasing because the decrease of tensile energy is greater than the increase of stretch [37].

Tensile Strength shows a lightly increase to a maximum value at around 35% RH and then this value starts to rapidly decrease at increased relative humidity. This phenomenon can be explained by the fact that the fiber-to-fiber bonding is weakened [37].

Tear is the property that is increasing in the interval used in the Figure 9. Nevertheless, after 80% at some RH value the curve will start to drastically decrease, because of the interfiber bonding disruption [37].

Folding endurance is a property which is highly influenced by the moisture content until around 65% and then it decreased. This property is influenced by different factors; thus, its analysis is complex [37].

Stiffness continually decreases with the increase of relative humidity. This is caused mainly by the increase of fiber flexibility [37].

Tensile Energy Absorption (TEA) is also impacted by the paper's moisture content. According to Szewczyk & Głowacki (2018), the increase of humidity will initially increase a paper TEA value, and eventually it will decrease it. Each paper has a specific maximum humidity value which provides the maximum TEA quantity [38].

According to the above-mentioned reasons, it is necessary to design packages that can withstand specific moisture contents, according to the product packed and the environment conditions it will meet, in order to guarantee its strength and quality.

2.3.10 Pigment Coating

Aqueous pigment coating is applied to paper by several methods such as spray coating, UV coating, curtain coating and so on. This coating type affects the paper properties as: improvement of printability; increase of the surface strength; the decrease of mechanical strength when compared to uncoated of the same grammage; and the decrease of stiffness when comparing paper of the same grammage [39].

The presence of pigment coating in a paper can affect its puncture resistance property. Once the existence of it can improve a paper's surface strength and mechanical strength, there is a potential that it would also increase its puncture resistance.

2.3.11 Polymer laminates and strength

All packages are required to accommodate the product and to the resist to any type of load that the product can suffer from its production until its consumption. In packaging, which consists of multilayers, the presence of plastic will contribute not only for the barrier properties, but also to improve the packaging's strength [30].

The density and the mass of the product packaged is a critical stress factor. Once the product's weight is higher than the laminates strength, it is possible that the package will be damaged. Therefore, it is necessary to design packaging that can withstand this load, for example by the addition of polymer laminates such as OPET (oriented polyester), which has high yield strength and is stiff [30].

3. PUNCTURE RESISTANCE

The measurement of puncture resistance is a type of testing standard which focus on the analysis of a sample's ability to resist being penetrated by an external object. During this process, force is applied against the sample at a predetermined and constant speed [40].

3.1 Paper properties and factors affecting them

Puncture resistance is the paper or paperboard resistance to penetration damages. A material with higher puncture resistance is stronger against these forces.

If the puncture load is strong enough a hole in created in the package (Figure 10). This damage should be avoided because this aperture will reduce the barrier properties, which can lead to product's loss of freshness and contamination. Furthermore, this will impact the appearance of the packaging [41].

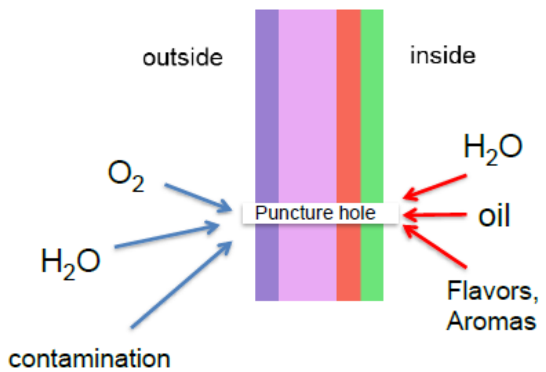


Figure 10. *Puncture hole in the package and its consequences [41].*

This property is essential in packaging because a package can be damaged by penetration parts from the products inside the packages (noodles, bones, frozen food), or during the transportation and handling [30,42].

The packaging material manufacturers have the responsibility to provide packages which have enough puncture resistance to ensure the safety and quality of the product inside. Therefore, each package type should be tested for puncture resistance using an appropriate machine [43].

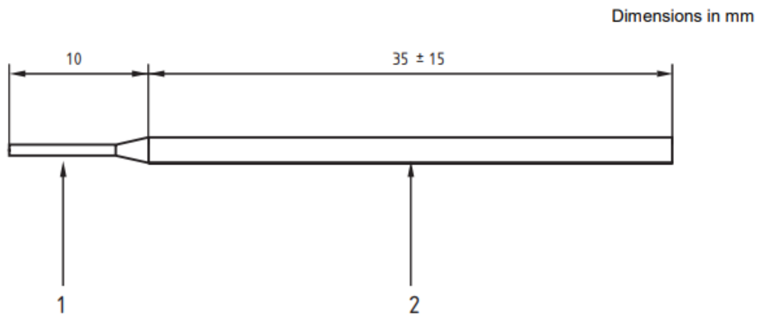
3.2 Puncture resistance variables

Several variables have influence on puncture resistance testing in packaging. The selection of them should be done in order to better emulate how puncture penetration damages happen in real life.

Theses variables selection is done according to the sample tested, which is paper in this work. The following topics will illustrate some of them.

3.2.1 Penetration probes

The penetration probe is the tool which pierces the sample material. It influences directly on the material's damage strength and its formats. Figure 11 illustrates the penetration probe format used in the standard *EN 14477:204 - Flexible packaging material, Determination of puncture resistance — Test methods* [44].



Key
1 Probe tip ∅ 0,8 mm ± 0,005 mm
2 Probe base ∅ 2,0 mm ± 0,010 mm

Figure 11. *Probe penetration standard shape [44].*

The probe shape can vary depending on the material being tested and its standard. Different shapes are shown on Figure 12 [42]. In this work both flat and hemispherical geometries were selected. The probe base is the same for every probe, but the tip will vary.

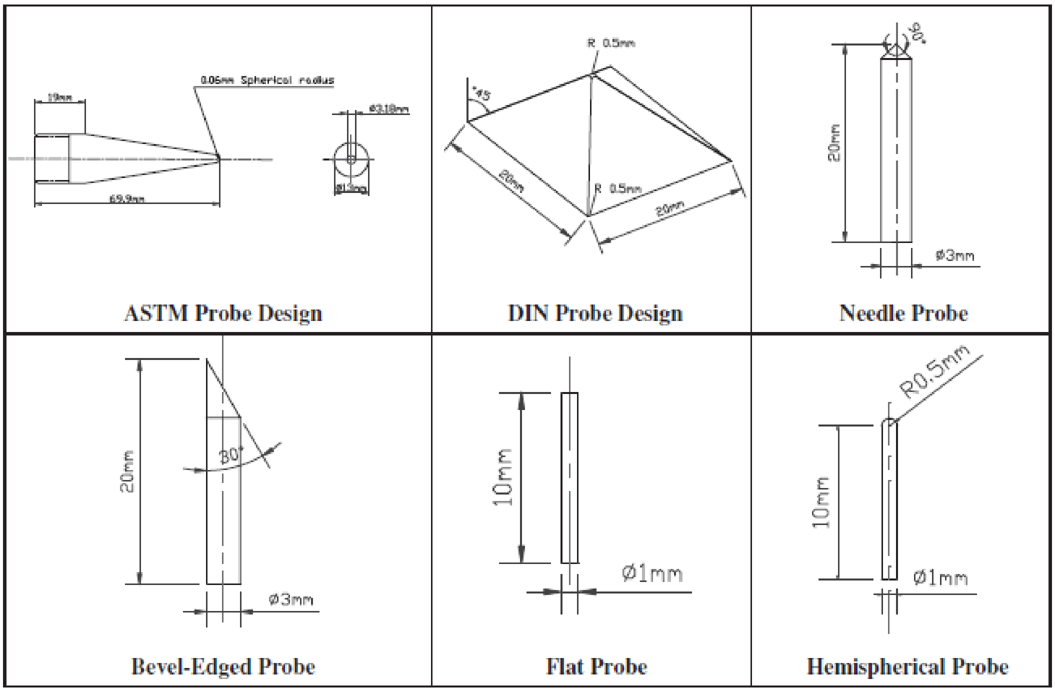


Figure 12. *Different penetration probes [42].*

The flat probe was chosen because it is the geometry used in the standard EN 14477:204. Even though this standard is not specific for testing the puncture resistance of paper, this was selected because there is not at the moment a standard which is paper specific for this type of testing. Also, this is the standard which tests a material that is more similar to paper.

Furthermore, the selection of the hemispherical probe is based on the article *Understanding Puncture Resistance and Perforation Behavior of Packaging Laminates*, by Lange et al. (2002). According to its writers, the hemispherical probe is a great alternative for testing packaging laminates. This type provides both damages shape like real life damages and reproducible results [42].

The tip shape will influence on the stress concentration during the puncture testing. The puncture resistance values are dependent on it. In this work, flat and hemispherical probes with different radius are used, each will have a different stress concentration and load distribution behavior. The sharper the tip, the higher the stress concentration caused. Rounded corner will reduce the stress concentration [45].

According to Sharma et al. (2004), to prevent geomembrane to be punctured, it is necessary to prevent it from the stress concentration of puncturing objects [46]. Similarly, in order to protect the paper used in packaging, it should be avoided it to be pierced by any penetrating objects. Particularly object which causes higher stress concentration on the substrate.

As shown on Figure 12, hemispherical probe has a tip shape which provides higher stress concentration than the flat probe. Therefore, it is expected that a paper is easier pierced by this hemispherical probe, thus it results in lesser puncture resistance, compared to the flat one.

3.2.2 Speed

The penetration rate speed varies according to the tested materials. This value is measured by mm/min. The tests can be characterized as slow puncture or high-speed puncture tests [30].

Tests performed at higher speed are commonly used for medical devices like suturing needles, and for sharp containers. On the other hand, tests at lower speed are used for films, geotextiles [47]. Paper is usually measured at slow speed.

The influence of speed on puncture resistance of some materials are somewhat limited. According to Lange et al. (2002), in their range speed tests and using a hemispherical probe, the rate testing for flexible material had small influence on the puncture resistance value [42].

3.2.3 Perforation angles

Usually the perforation angle is 90^{o} between the substrate and the probe, as in this research project. Different perforation angles can be selected in other to mimic different damages that a package can suffer in real life conditions.

3.2.4 Friction

Friction is known as the resistance to movement between two solid surfaces [48]. During the puncture resistance testing, the friction between the substrate's surface and the probe tip will work as a resistance to the penetration movement [49].

To perform the puncture resistance tests it is necessary to analyse the metal surface of the probe because friction changes if the metal surface is dirty or if it is oxidized. Commonly a polished metal surface causes less friction than a rough surface [16].

Different paper properties and processes in papermaking will change the paper's friction value. Normally coated paper has a distinct friction value than non-coated, and a paper with high humidity will have higher friction. Furthermore, the fillers presence, calendering and sizing can change the friction quantities [16].

3.2.5 Package properties

Puncture resistance is a material property. Therefore, the puncture resistance of a package is influenced by its composition.

3.2.6 Food packages

It is important to take in consideration the interaction between the packed food and the packaging material. Mass transport of organic and inorganic compounds, gases, water, and grease can impact the quality of the packed product [10], and the packaging.

This interaction can decrease the packaging's puncture resistance. If this happens, the packaging will be more susceptible to be damaged by external and internal forces.

3.3 Standard for measuring puncture resistance

All the puncture resistance experiments done in this thesis were performed accordingly with the EN 14477:2004 standard. This European Standard is used to determine the puncture resistance of flexible packaging materials.

4. MATERIALS

4.1 Paper samples

The papers tested in this thesis are listed in the following table (Table 2). The types used for each part of the testing are mentioned during the Results and Analysis section. Examples of all the papers samples are shown on Figure 13.

Table 2. *Paper samples tested.*

Paper	Grammage (g/m^2)	Bleached/ Unbleached	One Side Coated/Glazed	Company
1. ClearPack	62	Bleached	None	Arjowiggins
2. Flexi	62	Bleached	None	Arjowiggins
3. UPM Brilliant™ Pro	62	Bleached	Coated	UPM
4. Adv. Smooth White Strong	80	Bleached	None	Mondi
5. Adv. MG White Cote RB	47	Bleached	Glazed	Mondi
6. HS Bag Wgom	88	Bleached	Coated	Mitsubishi HiTec Paper
7. Bag Wgom 70	70	Bleached	Coated	Mitsubishi HiTec Paper
8. PackPro 7.0 Rotogravure	70	Bleached	Coated	B&B Labels and Flexpack
9. Adv. MG Coating	40	Unbleached	Glazed	Mondi
10. Adv. MF Z	50	Unbleached	None	Mondi
11. Adv. MG Kraft	40	Unbleached	Glazed	Mondi
12. Adv. MG Kraft	80	Unbleached	Glazed	Mondi
13. Adv. Formable	100	Unbleached	None	Mondi
14. Adv. Formable	130	Unbleached	None	Mondi

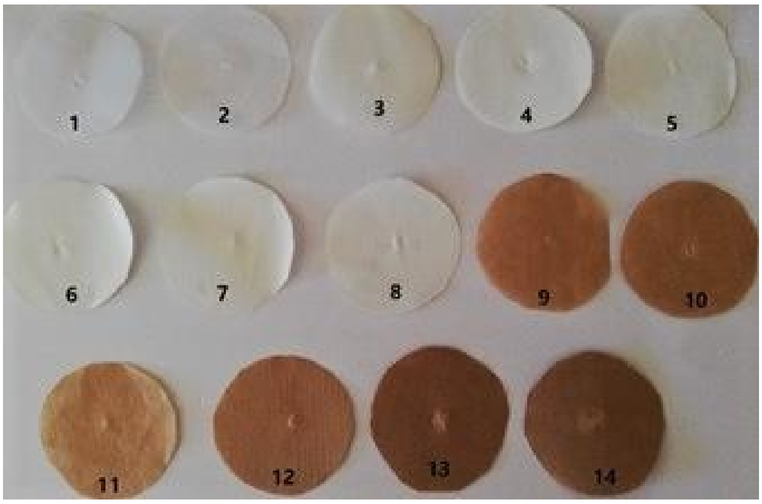


Figure 13. *Samples tested.*

4.2 Paper samples

4.2.1 Puncture resistance testing assembly

The puncture tests were performed using a universal testing machine – Instron 5967 (500N load cell). The experiments were possible after adapting this machine to this type of testing.

In order to do so, it was designed and manufactured a sample holder, a probe holder and penetration probes, as explained next. Figure 14 depicts the schematic presentation of the testing arrangement.

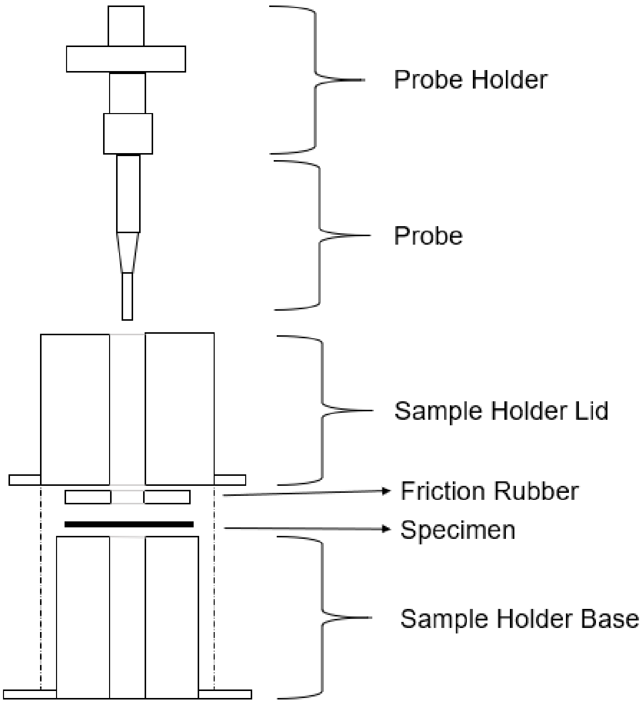


Figure 14. *Testing arrangement schematic presentation.*

4.2.1.1 Sample holder

The sample holder is the tool by which the sample is accommodated still during the experiments. It consists of a base and a lid; the later has a hole for the penetration of the probe.

The following picture illustrates the sample holder system. Part A shows the sample holder base with the sample on its designed position. Part B illustrates the sample holder with the lid on. The lid has a rubber inside to prevent the sample from moving during the experiments. Also, the lid is hold tight with the use of levers.

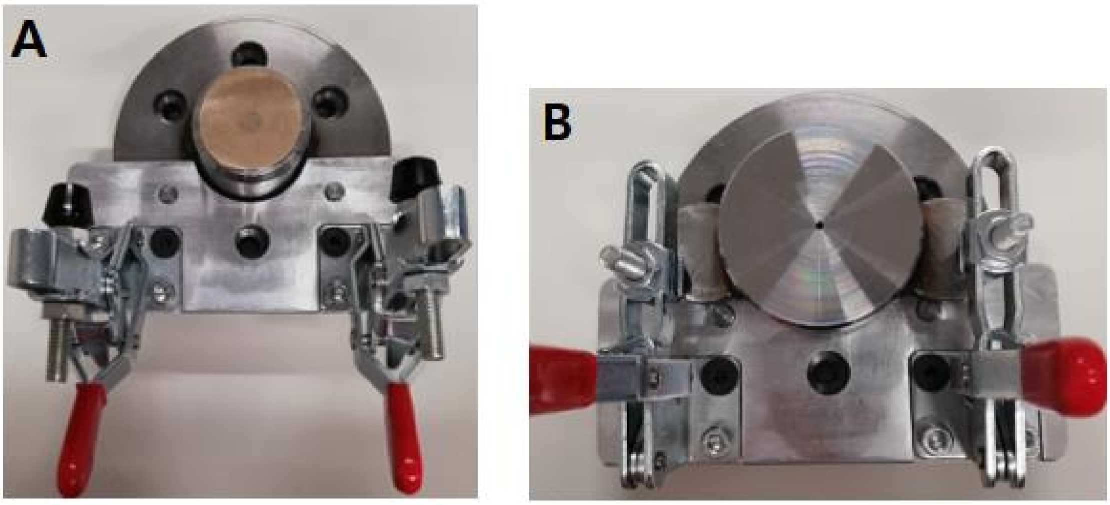


Figure 15. *Sample holder and a sample [A], Sample holder with lid on top [B].*

4.2.1.2 Probe holder

The probe holder holds the penetration probes tight during the experiments as shown on Figure 16.

Figure 16*. Probe holder and a penetration probe.*

4.2.1.3 Penetration probes

In this thesis, five different probes were manufactured and used. Each one has the same shaft dimensions, but their tip changes, as shown in the Table 3.

Table 3. *Probes specifications.*

Probe	Tip Geometry	Tip radius	Material used	Hard/Soft tip
A	Flat	0,8 mm	Steel	Hard Tip
B	Hemispherical	0,8 mm	Steel	Hard Tip
C	Hemispherical	1,6 mm	Steel	Hard Tip
D	Hemispherical	2,4 mm	Steel	Hard Tip
E	Hemispherical	2,4 mm	Steel + Urethane (Shore A 40)	Soft Tip

Probes A were manufactured accordingly to the standard EN 14477:204 (Figure 7). Probes B, C and D are hemispherical probes, with tip diameter value increasing in 0,8 mm from the previous probe.

The probe E, Soft tip, has a shaft made of steel and it is covered by a soft material, which is Urethane (Shore A 40). The addition of the soft material was done in order to imitate the impact of a packed chocolate bar into its packaging. Its manufacture process is further explained in the following paragraphs.

Initially, in order to select the soft material, the hardness of Fazer's Blue chocolate and Nestle's Kit Kat was measured in Shore A scale. A durometer was used to measure their hardness, in different positions for one minute. The values of hardness during the experiments are shown in Table 4.

Table 4. *Chocolates hardness measurement.*

Chocolate	Position	Hardness (Shore A)		
		15s	**30s**	**60s**
Fazer Blue	Bottom	48,95	48,6	48,3
		50,25	49,25	49,5
	Side	44,5	44	44
		44,95	44,4	44,1
Kit Kat	Bottom	34,2	34,1	33,95
		18,5	18,5	19,75
	Side	23	22,95	22,98
		13,8	13,65	13,5
	Surface	32,95	32,95	32,9
		15,1	15,1	15,1

The hardness measured for both chocolates is different, independent of the side tested. Fazer chocolate varied between 40-50 Shore A Hardness. However, Kit Kat values varied a lot depending on the side tested, once there are biscuits inside it. Kit Kat hardness reached maximum value of 34.

Finally, after the hardness measured, it was chosen a Urethane Shore A 40 as soft material because this hardness value is similar to the values obtained from both chocolates tested.

A mold made of silicone was built using the Hemispherical Probe D (2,4 mm). With this mold, the soft probe was cast using a shaft made of steel and the Urethane Shore A 40. This probe was cured, and then the soft probe E, which has the same dimensions of Probe D, was manufactured for testing.

4.2.2 Tensile Tester

A tensile tester -Hounsfield H10KM (100N load cell)- was used to measure the tensile properties of the paper samples. The properties measured were: Tensile Strength; Tensile Index; Stretch/Strain; Tensile Energy Absorption (TEA), and Tensile Energy Absorption Index. This was necessary for the study of the correlation of these properties to the puncture resistance.

4.2.3 Shore Hardness Tester

A Shore Hardness tester was used for testing the hardness of different chocolate brands: Fazer Chocolate; and Nestlé Kit Kat. This measurement was needed in order to help the selection of the material for the soft probe as previously mentioned.

4.2.4 Moisture Analyzer

A Halogen Moisture Analyzer HR 733 was used to determinate the moisture content of the paper samples used during the experiments. They were conditioned in two different conditions: 50% RH and 23^{o}C; 75% RH and 23^{o}C.

5. METHODS

5.1 Puncture Resistance

All paper samples were tested for puncture resistance according to the Standard EN 14477:204. This experiment measures the puncture resistance, maximum load, and maximum strain of the paper sample. The testing speed was 10 mm/min.

In order to perform the tests, the samples were previously conditioned at two different conditions: 50% RH and 23ºC (standard condition); 75% RH and 23ºC. These different conditions were selected in order to study the influence of the humidity on puncture resistance.

The tests were performed using a sample holder and a universal tensile tester attached with probe holder and a probe. The penetration probe attached into the machine moves, at constant speed, until it perforates the sample (that is fastened under an aperture in the lid). Both force and the displacement needed to perforate the sample are recorded [34].

The following figure demonstrate a typical curve obtained from this experiment – Extension (mm) vs. Load (N). It depicts the Puncture Resistance (PR), Maximum Load (ML) and Maximum Strain (MS).

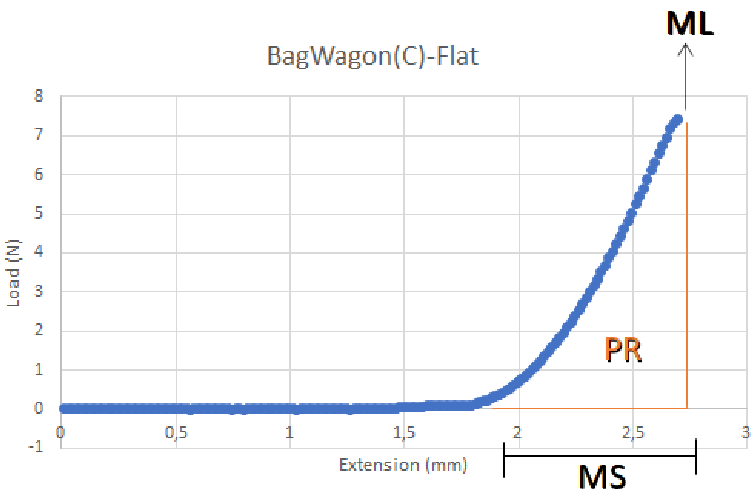


Figure 17. *Example of curve obtained from the Puncture Resistance experiments.*

Puncture Resistance is a function on the ML and MS, which is calculated as the area (integral) below the graph. Maximum load is the maximum load obtained during the experiment. Maximum strain is the difference between the final extension and the extension point where the load starts to increase.

5.2 Tensile Testing

All papers were tested for their tensile properties according to the *Standard ISO 1924-3: Determination of Tensile Properties*. The following properties were tested: Tensile Strength; Tensile Index; Strain at Break; Tensile Energy Absorption (TEA); and Tensile Energy Absorption Index.

In order to perform the tests, all samples were conditioned at 50% RH and 23ºC during the minimum time mentioned in the standard. All the samples were testes in Machine Direction (MD) and Cross Direction (CD).

The tests consisted of the elongation of a test piece until it brakes (Figure 18), at constant elongation rate of 100mm/min using a specific testing machine, which records during the experiment the tensile force and the elongation [50].

***Figure 18**. Tensile Tests.*

The data format of the experiments is illustrated in Figure 19. It is an Elongation vs. Force curve, from which different tensile properties can be measured.

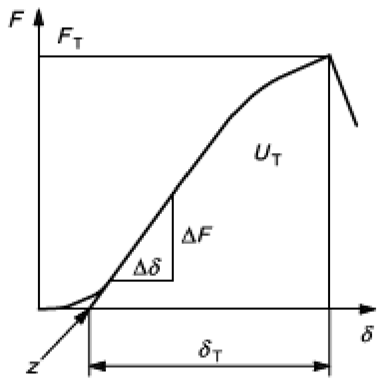


Figure 19. *Typical Elongation vs. Force curve with quantities measured [50].*

Where: F is tensile force (N); δ elongation (mm); Z is the point that the tangent of the curve – where a slope that is equivalent to the curve's maximum slope - intersects the δ axis; F_T is the maximum tensile Force (N); elongation at break (mm); U_T is the area below this curve (mJ).

The following sections describe more about the properties measured during these experiments according to ISO 1924-3. Geometric mean was retrieved from Khan, K. (2011) - *Development and evaluation of a puncture strength test method for sterilization paper* [19].

5.2.1 Tensile Strength

It is the highest tensile force by unit width that the sample can resist before it breaks. It is measured as following:

$$\sigma_T^b = \frac{F'_T}{b} \text{ (1)}$$

σ_T^b: Tensile Strength (kN/m)

F'_T: Mean maximum tensile force (N)

b: Width of test piece (mm)

5.2.2 Tensile Index

It is the tensile strength divided by the paper's grammage. It is calculated as:

$$\sigma_T^W = \frac{1000\sigma_T^b}{w} \text{ (2)}$$

σ_T^W: Tensile Index (KNm/kg)

σ_T^b: Tensile Strength (kN/m)

w: Grammage (g/m^2)

5.2.3 Strain at break (Stretch)

It is the strain obtained from the maximum tensile force. As follows:

$$\varepsilon_T = \frac{100\,\delta'_T}{l} \quad (3)$$

ε_T : strain at break, as the percentage of the test initial length

δ'_T: mean elongation at break (mm)

l: initial test piece length (mm)

5.2.4 Tensile Energy Absorption (TEA)

It is the quantity of energy per unit surface area of a test's piece when this piece is strained to the upmost tensile force.

$$W_T^b = \frac{1000\,U'_T}{bl} \quad (4)$$

W_T^b: Tensile Energy Absorption (J/m^2)

U'_T: Mean area below the elongation-force curve (mJ)

b: Initial test piece width (mm)

l: initial test piece length (mm)

5.2.5 Tensile Energy Absorption Index

It is the Tensile Energy Absorption divided by the paper's grammage

$$W_T^W = \frac{1000 W_T^b}{w} \quad (5)$$

W_T^W: Tensile Energy Absorption Index (J/kg)

W_T^b: Tensile Energy Absorption (J/m^2)

w: Grammage (g/m^2)

5.2.6 Geometric mean

Geometric mean of previous properties, can be calculated with its MD and CD readings

$$Pgeo = \sqrt{P_{MD} * P_{CD}} \quad (6)$$

$Pgeo$: Geometric mean

P_{MD}: Mean values of Machine Directions readings

P_{CD}: Mean values of Cross Directions readings

5.3 Hardness testing

The hardness of two different chocolates – Fazer chocolate and Nestlé Kit Kat–, were analyzed using a Durometer, measured by a Shore A scale. The Shore A is a scale commonly used to measure soft polymers and elastomers [51]. Here it was used for chocolate bar.

The Durometer uses an indenter, which is loaded by a calibrated spring. The determination of the hardness was determined by the penetration depth of the Shore A indenter into the material, under a specific load, as show below [51]. The hardness tests were done in different chocolates sides. Also, it was measured in during different penetration times.

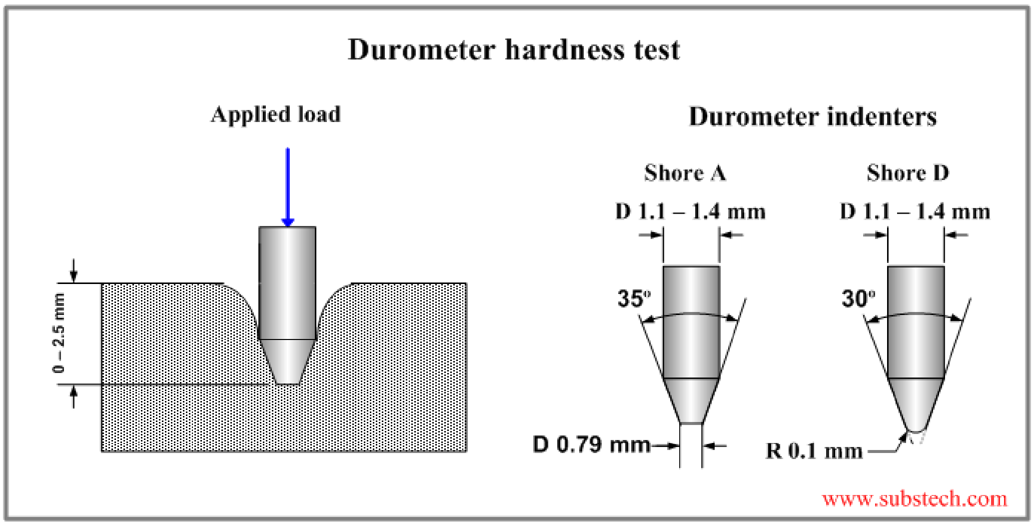


Figure 20. *Typical Elongation vs. Force curve with quantities measured [51].*

5.4 Moisture content measurement

The moisture analyzer, Halogen Moisture Analyzer HR 733 (Figure 21), was used to measure how much the paper's sample moisture content changed while being conditioned in two different conditions: 50% RH and 23°C (standard condition); 75% RH and 23°C.

Figure 21. Halogen Moisture Analyzer HR 733 *[52].*

The operation of this machine is done according to the thermogravimetric principle. At the beginning of the measurement this analyzer will measure the sample's weight, then the sample is fast heated by the built-in halogen dryer unit which vaporizes the moisture. Throughout the drying stage, the equipment constantly measures the sample's weight, showing the decrease of moisture. After the drying process is done, the final result of the moisture content is shown [53].

6. OBJECTIVES

The objectives of this research project are listed below:

1. To design and manufacture the equipment needed for the puncture resistance tests.
2. To test and analyze how different parameters, such as probe design and moisture content, affect the puncture resistance of different papers.
3. To determinate how the puncture resistance correlates with other mechanical properties with different papers.
4. To analyze possible causes for differences in puncture resistance of the paper’s samples used.

7. RESULTS AND DISCUSSION

7.1 Puncture Resistance Tests

Several different puncture resistance experiments were performed during this thesis. The following topics present different conclusions obtained with their analysis.

7.1.1 Probe Design

Five different probes were used in this research project to study puncture resistance of different papers. Considering that puncture resistance is a function of maximum load and maximum strain, these were also analyzed. The papers tested in each section are either mentioned or illustrated in the graphics.

The following topics studies how different probes influenced on the paper's puncture resistance. In order to be more illustrative, it was decided to group the probes in a common characteristic.

7.1.1.1 0,8mm Probes: Flat (Probe A) & Hemispherical (Probe B)

In this section, it will be discussed the influence of the probe geometry, specifically the tip part. Probe A and B both have the tip diameter of 0,8 mm.

The following graphs compares how papers' values of maximum load, maximum strain, and puncture resistance varied between the probes' designs.

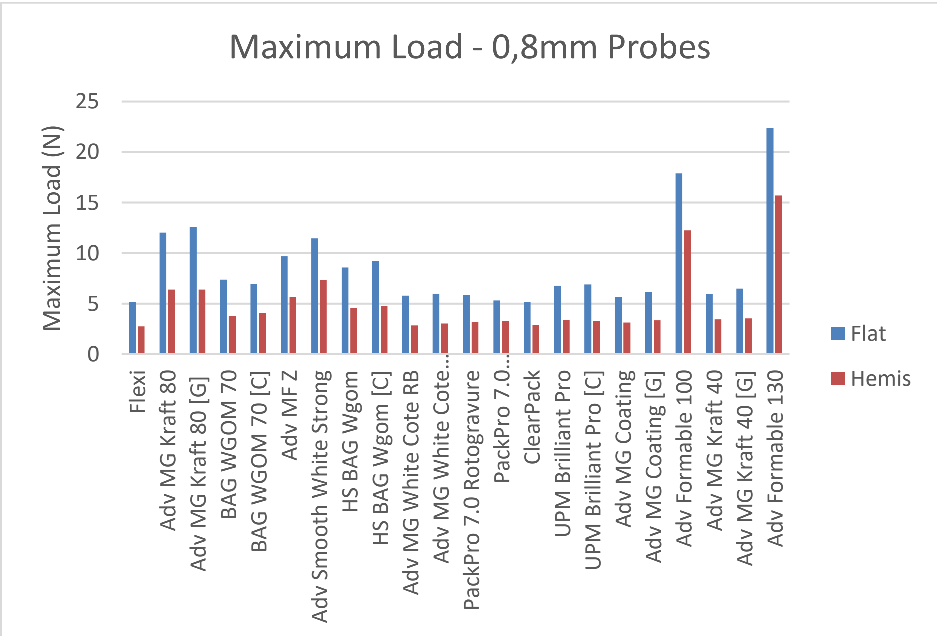


Figure 22. *Maximum Load obtained using 0,8mm Probes.*

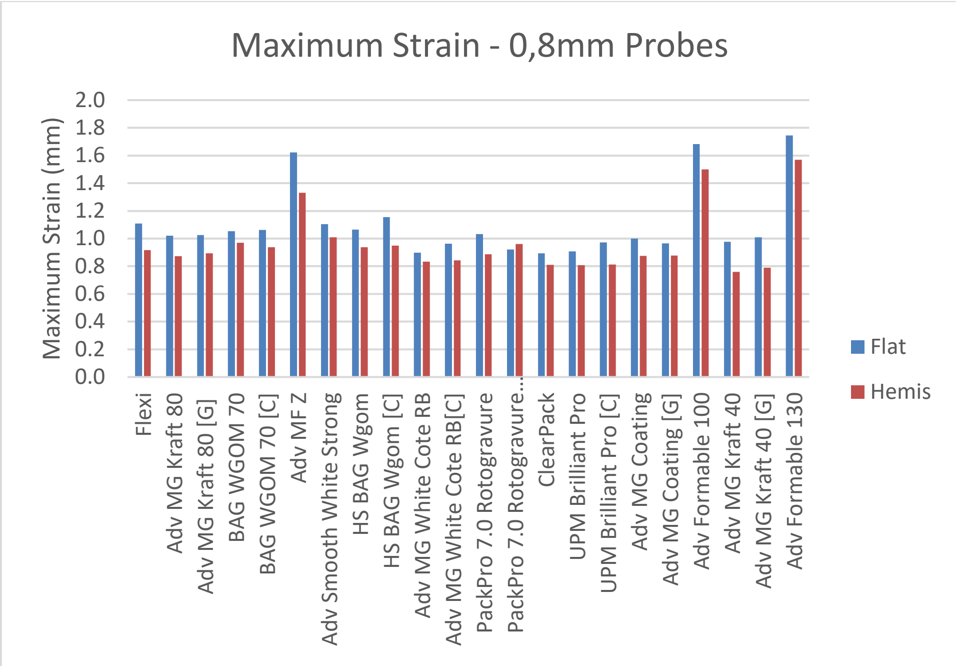


Figure 23. *Maximum Strain obtained using 0,8mm Probes.*

Figure 22 and Figure 23 respectively shows the comparation of different papers' maximum load and maximum strain. It is clear that for both measurements, the flat probe provides higher values than the hemispherical probe. Furthermore, the difference between both probes' measurements is higher for the maximum load than for the maximum strain.

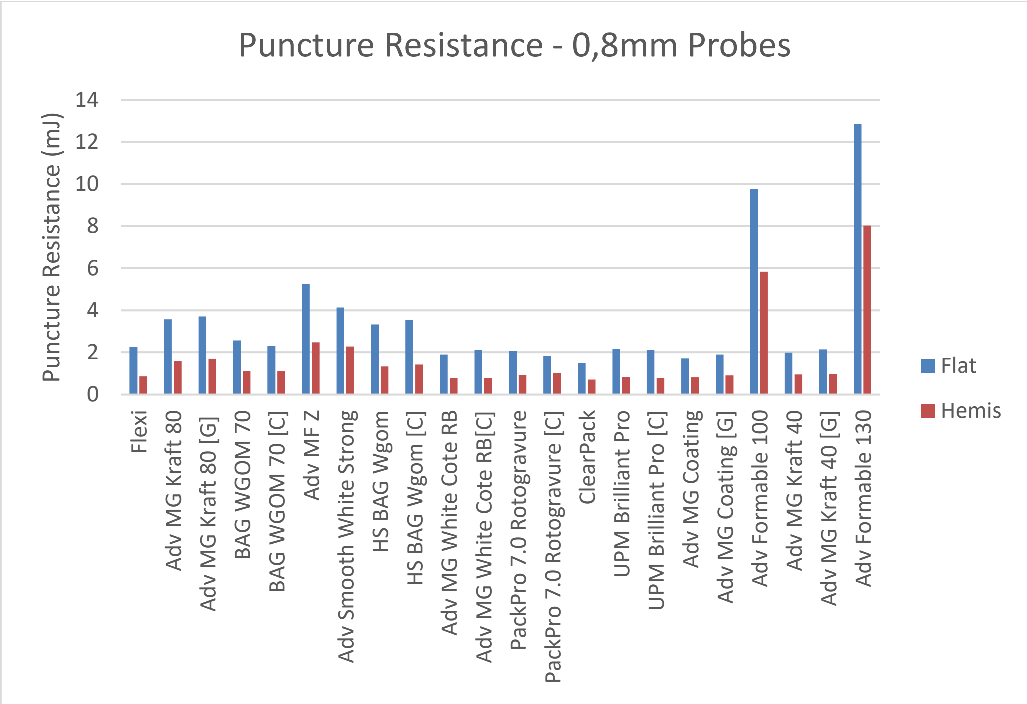


Figure 24. *Puncture Resistance obtained using 0,8mm Probes.*

Figure 24 illustrates that papers' puncture resistance is higher while using the Flat probe. This result is a consequence of the higher values obtained for the maximum strain and maximum load, once puncture resistance is a result of them.

The Hemispherical probe causes a higher stress concentration on the paper compared to the Flat probe. A higher concentration stress facilitates the paper to fail during the experiments. Therefore, the puncture results values are smaller using a hemispherical probe.

7.1.1.2 Hemispherical Probes: 0,8mm (Probe B); 1,6mm (Probe C); 2,4mm (Probe D)

The influence of the tip size will be discussed in this section. Advantage Formable 100 $g/^2$, at 50% RH, was selected to illustrate how these probes influences on a paper's puncture resistance.

Different graphs were plotted, and it was added the tendency line (trend line) and the coefficient of determination (R^2) to all graphs, to quantify any correlation.

The analytical tool known as trend line is used in graphs to check if the data plotted has some pattern [54]. After its addition, the coefficient of determination can be calculated, which better quantify the relation between data.

The coefficient of determination (R^2) is commonly used to analyze how much the variability of one specific factor will be caused by its correlation with a different factor [55]. In this case, it was used to analyze how much the tested values correlates with different probes.

This determination is measured as value in between 0,0 and 1,0. A value closer to 0 indicates that this model does not efficiently model the data. However, values close to 1 illustrates that this is a very efficient model to forecast results [55].

First, it will be analyzed the differences in this paper's maximum load using these probes. Figure 25 illustrates these values, error bars, tendency line and R^2.

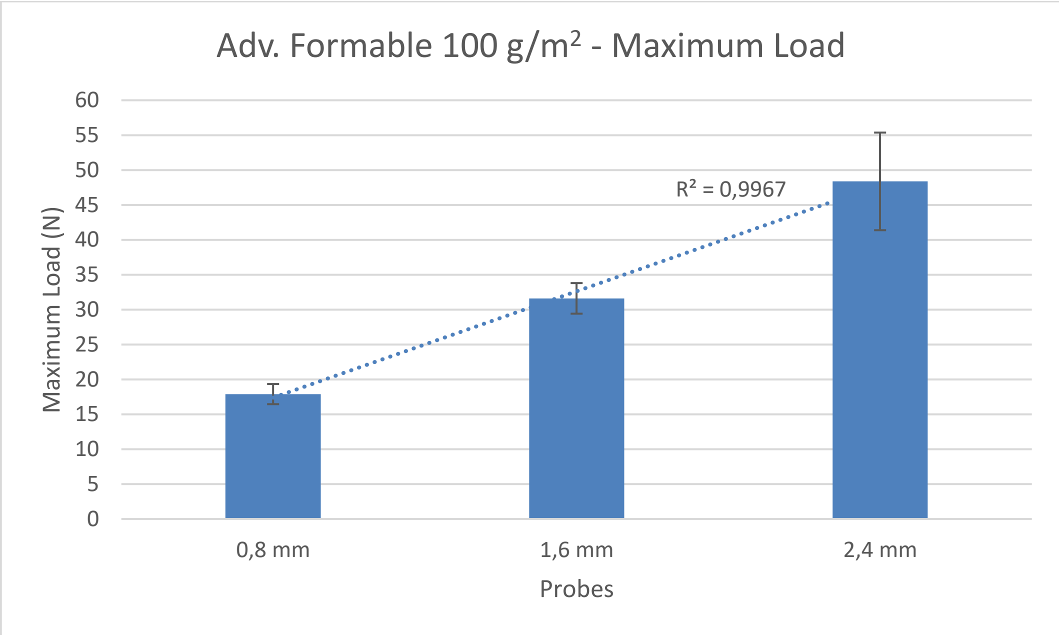


Figure 25. *Maximum Load Comparation – Hemispherical Probe.*

From Figure 25, it is noticeable that the increase of the probe's tip diameter will cause higher maximum load values. Also, there is a direct proportion between the probe size and the ML values. The R^2 value close to 1 proves that this is a good model to forecast this paper's maximum load values obtained with other hemispherical probes sizes.

Furthermore, it is noticeable that the standard deviation increases with the tip size. One reason for this phenomenon is that the increase of area will also raise the probability for weak points, which causes higher variance of results.

The following graph illustrates how maximum strains values varied between the probes used.

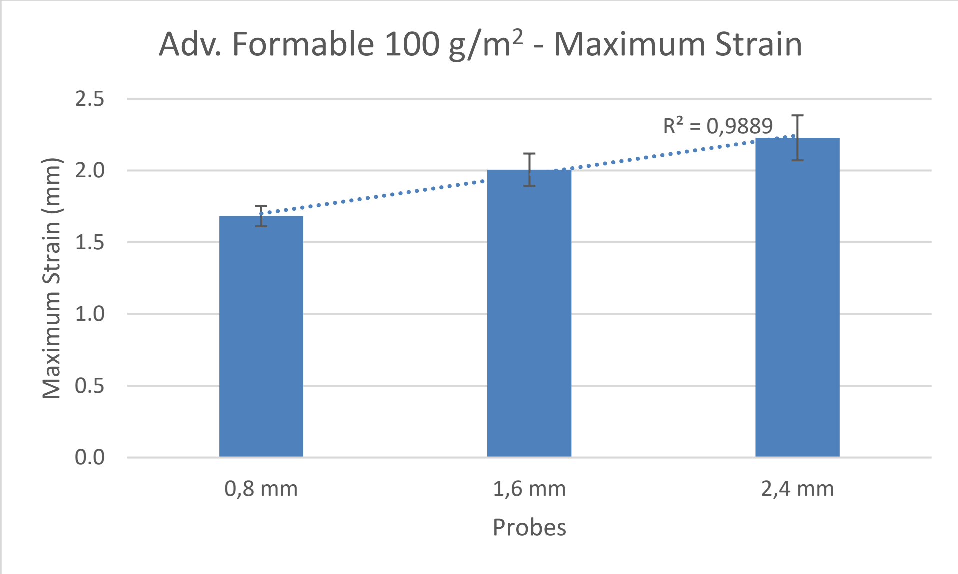


Figure 26. *Maximum Strain Comparation – Hemispherical Probe.*

Figure 26 depicts that the maximum strain values increases when the probe size also increases. There is not a direct proportion between the tip dimension and the MS values. However, the R^2 value close to 1, illustrates that this correlation is good and can be used to estimate this paper MS values while using different hemispherical tips dimensions.

Lastly, it will be analyzed the differences in this paper's puncture resistance values using these probes.

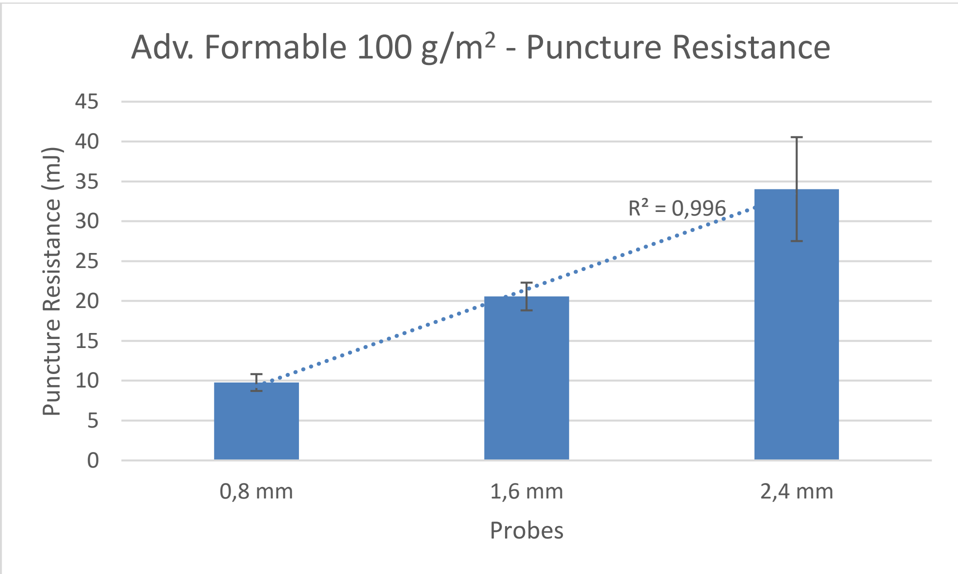


Figure 27. *Puncture Resistance Comparation – Hemispherical Probe.*

Figure 27 illustrates that puncture resistance increases with the increment of hemispherical probe tip. This was expected because both MS and ML also increased. There is a direct proportion between the tip dimension and the PR values. Finally, the R^2 value proves that this is a good model to forecast PR values for this paper using hemispherical probes.

Every tested paper, at 50% RH, had its results following the same behavior. Both maximum load and puncture resistance presents direct proportion with the tip dimension. However, there is not direct proportion while comparing maximum strain and tip dimension.

Furthermore, the measured values of maximum load, maximum strain and puncture resistance increased with the increase of the tip dimension. This can be justified once a bigger tip causes a smaller stress concentration on the paper, which collaborates for it to withstand higher loads before being pierced.

Lastly, the analysis of R^2 illustrates that these values will follow similar pattern, while using different tips sizes. This contributes to the estimate how different hemispherical probes' tip would affect the measurement.

7.1.1.3 Hemispherical Probes: 0,8mm (Probe B); 1,6mm (Probe C); 2,4mm (Probe D)

The influence of the probe material will be discussed in this section, where a hard tip probe will be compared with one soft tip. In this, three different papers were used for the analysis.

Figure 28 depicts the difference in behavior of the Extension vs. Load graphs of ClearPack paper, which is similar to all papers tested.

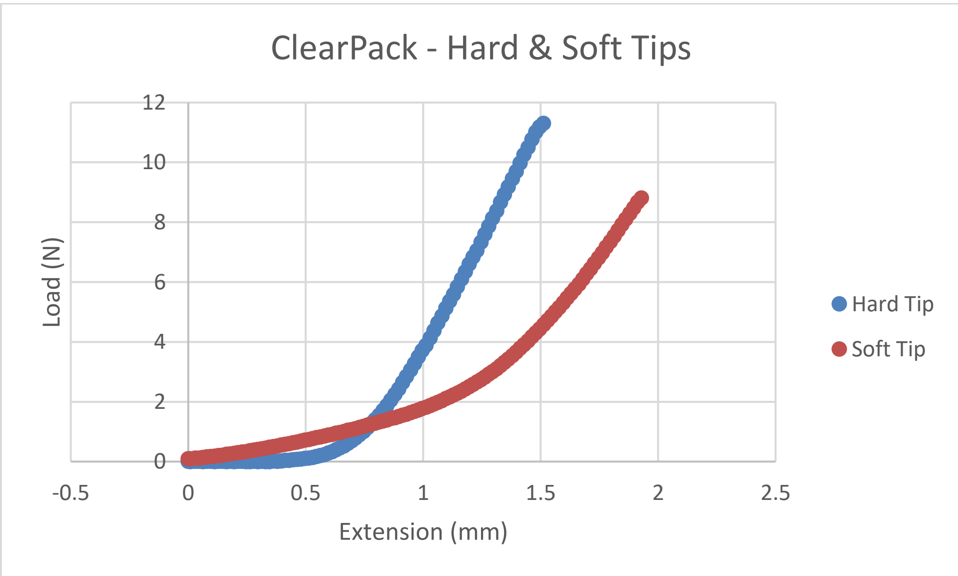


Figure 28. *Extension vs. Load curves – Hard & Soft tips.*

The hard probe has a delay before applying effective load to the paper, but when it does the increase of load is linear, reaching a higher number without extending the paper as much.

On the other hand, the soft probe already starts applying load to the paper as soon as the contact between both starts. The load applied is not as high nor increases rapidly as the hard tip, which can be justified by the fact that the soft probe will be deformed when in contact with the paper. As the load applied does not increase abruptly, it permits the paper to extend more before its failure.

The following graphs from Figure 29 and Figure 30, respectively illustrates the Maximum Load and the Maximum Strain of the tested papers.

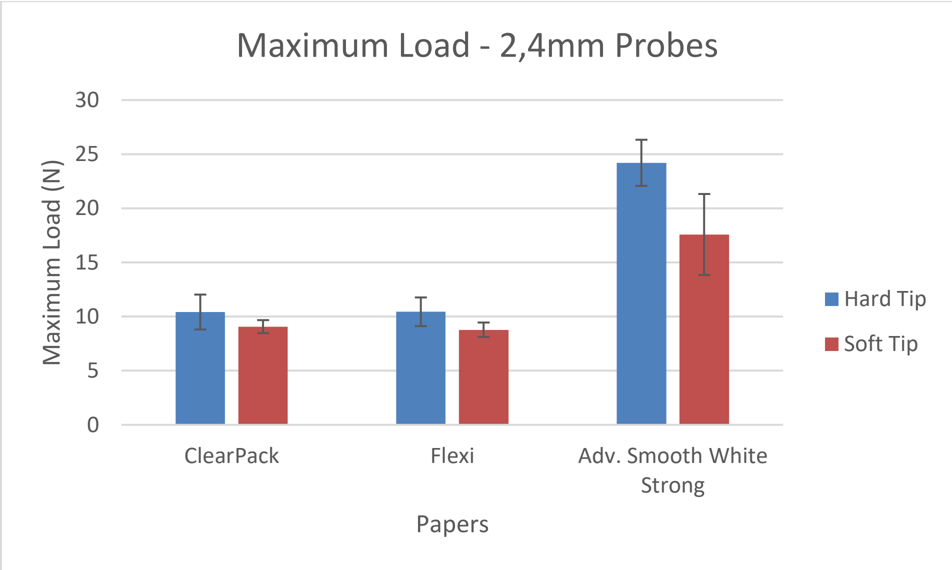


Figure 29. *Maximum Load – 2,4mm Probes.*

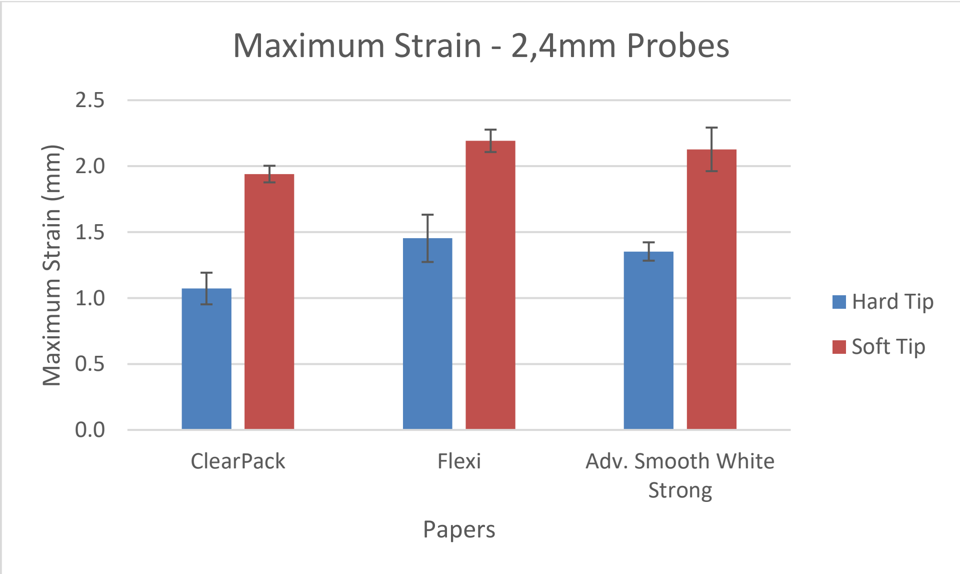


Figure 30. *Maximum Strain – 2,4mm Probes.*

As above-mentioned, the difference in behavior of the probes' Extension vs. Load graphs influenced on the paper's measured values. Figure 29 depicts that the maximum load is higher while using the hard probe, but this difference is not as high. However, Figure 30 illustrates that the maximum strain varied more between them, with the soft tip providing higher value.

Finally, Figure 31 compares papers' puncture resistance while using these probes.

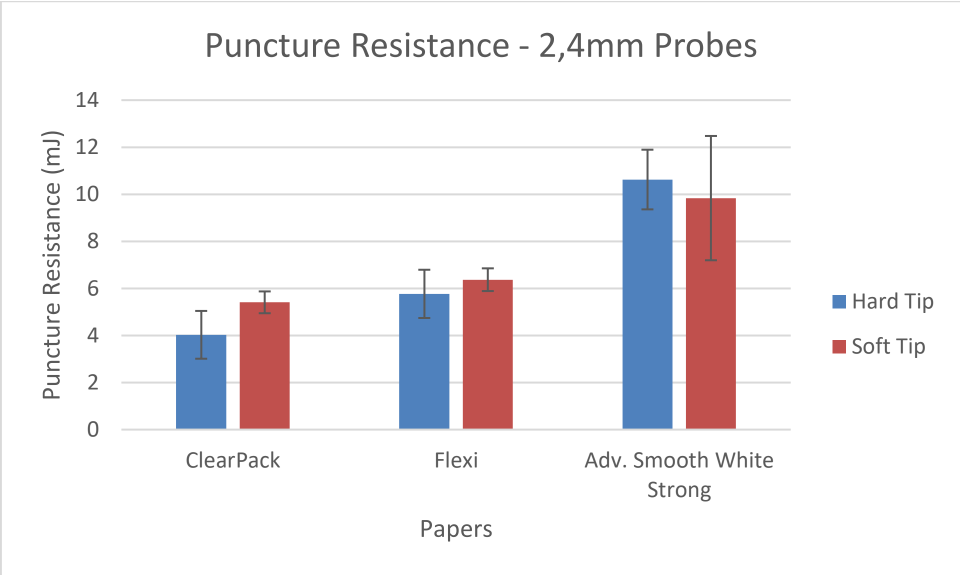


Figure 31. *Puncture Resistance Comparation – 2,4mm Probes.*

Figure 31 illustrates that paper's puncture resistance, did not significantly change while using a hard or a soft tip. This can be justified while taking in consideration that the stress concentration of both probes on the paper would be the same, once their dimensions are equal. The material type will only affect more significantly a paper's maximum strain.

The soft probe was manufactured to study how a product, in this case chocolate bars, affects its package, by perforation attacks. It seems that the product's material does not have a higher influence than the shape of this impact, in puncture resistance.

Higher amount of papers should be analyzed in order to better understand these differences. However, the soft probe was damaged after being tested with other papers. This implies that the soft material chosen was not hard enough to test all the paper types in this research project.

7.1.2 Puncture Resistance and Mechanical Properties

One of the objectives of this thesis is to study the correlation of puncture resistance with paper's mechanical properties.

In order to do so, first it was calculated the tensile properties (both directions) of the tested papers. Also, it was measured the geometric means values of these properties, which is commonly done in order to compare papers with dissimilar fiber anisotropies [56]. All measured values are attached at the Appendix.

After these measurements, it was possible to study the tensile properties (geometric means) relationship with puncture resistance. Graphs of Puncture Resistance and Tensile Properties were plotted to analyze if there is any correlation. In order to study and quantify any possible correlation it was added the tendency line and the coefficient of determination (R^2) to all graphs.

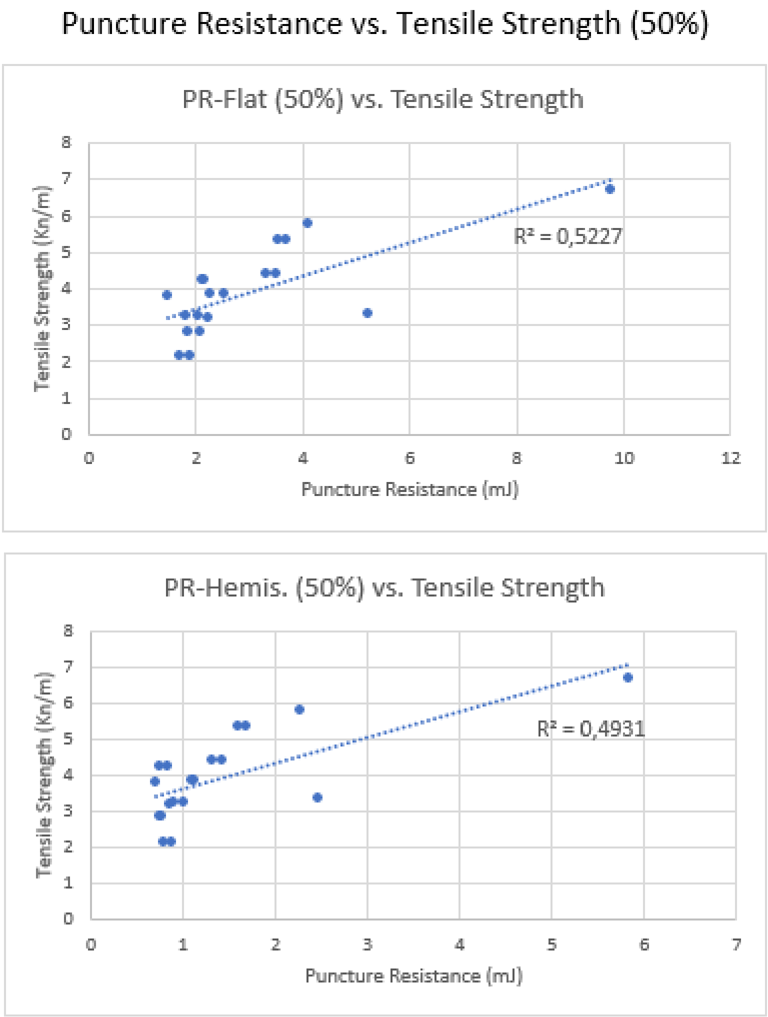


Figure 32. *Puncture Resistance at 50% RH (Flat & 0,8mm Hemispherical Probes) vs. Tensile Strength.*

Figure 32 illustrates Tensile Strength vs. Puncture Resistance of papers conditioned at 50% RH. The analysis of the coefficient of determination demonstrates that there is some correlation.

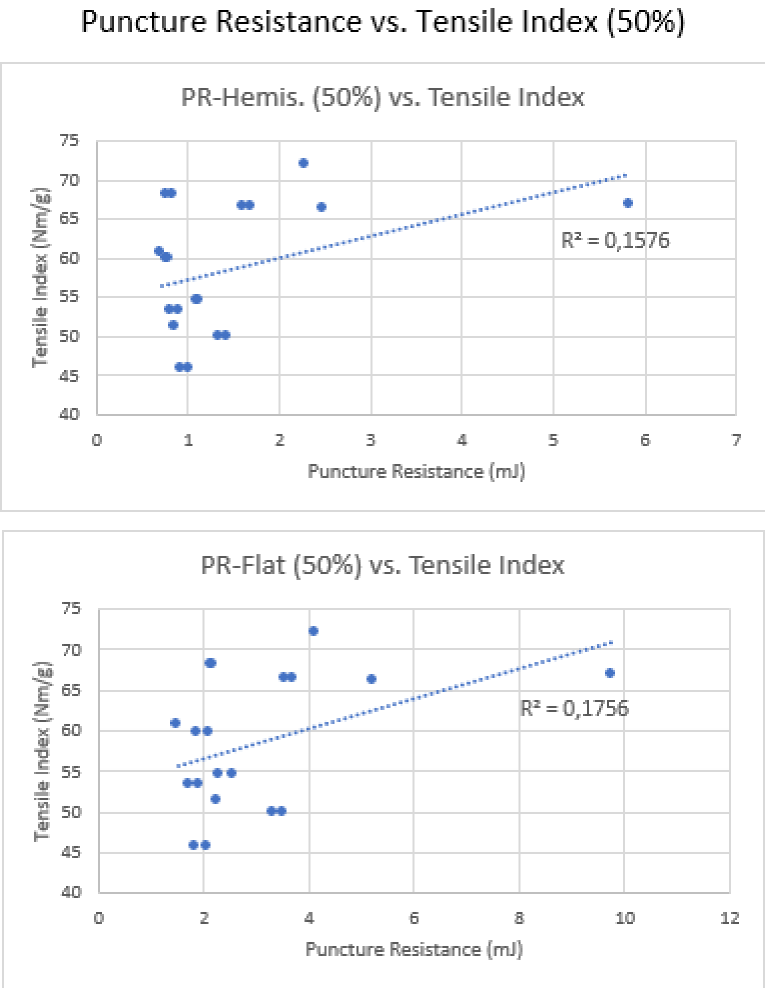


Figure 33. *Puncture Resistance at 50% RH (Flat & 0,8mm Hemispherical Probes) vs. Tensile Index.*

Figure 33 illustrates Tensile Index vs. Puncture Resistance of papers conditioned at 50% RH. The analysis of R^2 indicates that there is not any correlation between these two properties.

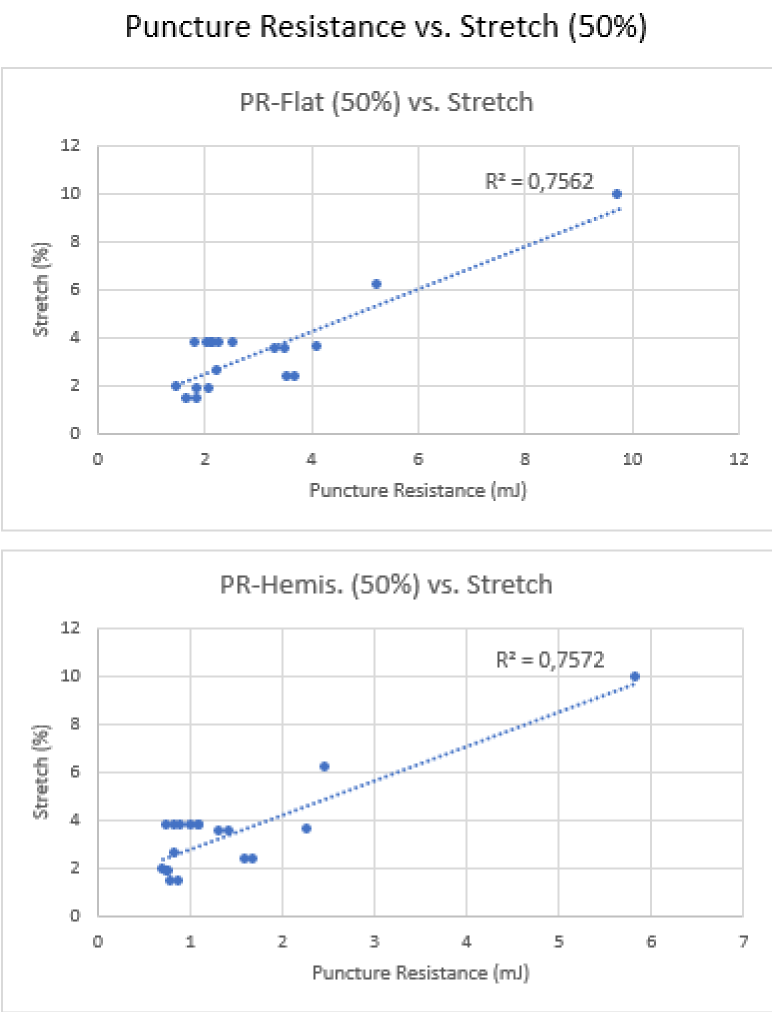


Figure 34. *Puncture Resistance at 50% RH (Flat & 0,8mm Hemispherical Probes) vs. Stretch.*

Figure 34 illustrates Stretch vs. Puncture Resistance of papers conditioned at 50% RH. The analysis of the coefficient of determination proves that there is correlation between both properties.

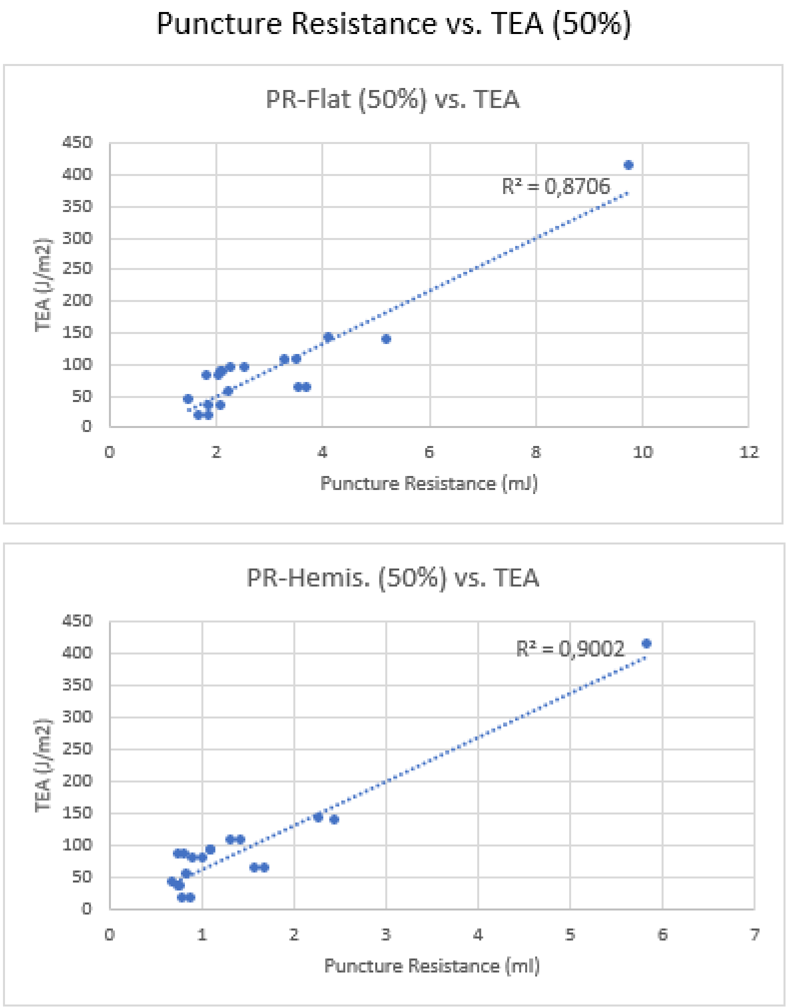


Figure 35. *Puncture Resistance at 50% RH (Flat & 0,8mm Hemispherical Probes) vs. Tensile Energy Absorption (TEA).*

Figure 35 illustrates TEA vs. Puncture Resistance of papers conditioned at 50% RH. The analysis of the coefficient of determination proves that there is correlation between them.

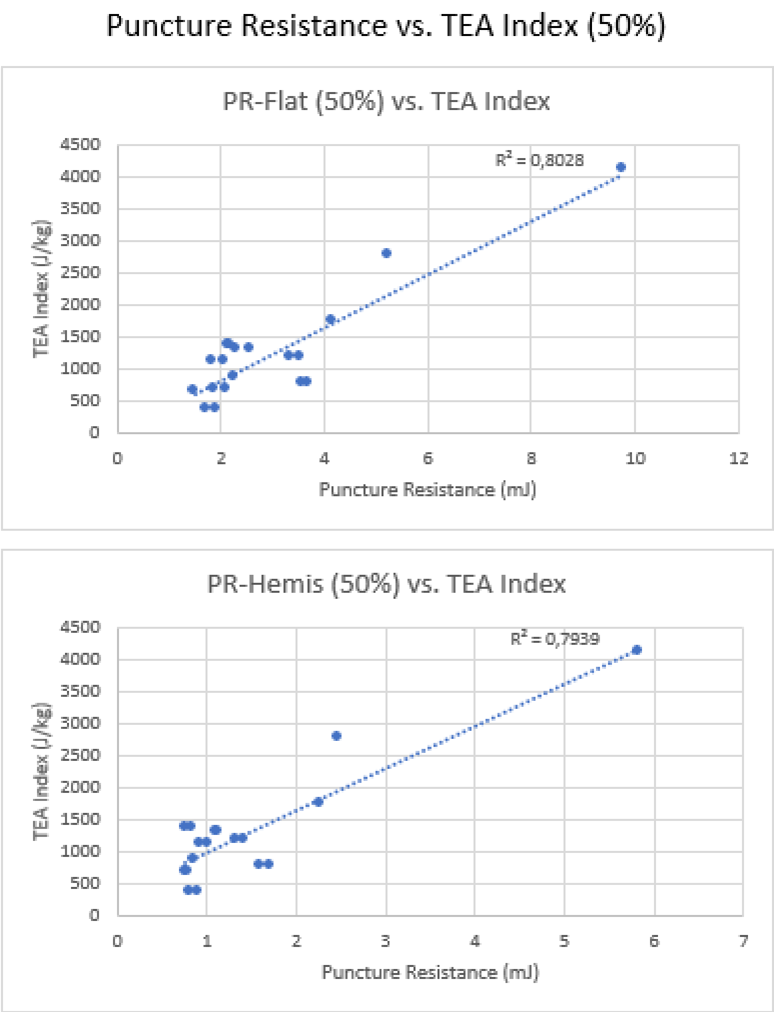


Figure 36. *Puncture Resistance at 50% RH (Flat & 0,8mm Hemispherical Probes) vs. TEA Index.*

Finally, Figure 36 illustrates TEA Index vs. Puncture Resistance of papers conditioned at 50% RH. R^2 demonstrates that there is correlation between these properties.

Similarly, this analysis was also done with paper's conditioned at 75% RH. The coefficient of determination of all graphs are shown in Table 5.

Table 5. *R^2 Summary of correlations.*

Property	**RH**	**Probe**	
		Flat	**Hemispherical**
Tensile Strength	50%	0,52	0,49
	75%	0,55	0,49
Tensile Index	50%	0,18	0,16
	75%	0,15	0,20
Stretch	50%	0,76	0,76
	75%	0,71	0,72
TEA	50%	0,87	0,90
	75%	0,88	0,82
TEA Index	50%	0,80	0,79
	75%	0,75	0,8

The Table 5 illustrates that neither the probe used, nor the relative humidity significantly changed the relations between puncture resistance and tensile properties. This is a good indication that these correlations would have similar values, despite the use of different probes and RH conditions.

Tensile Strength proved to have correlation with Puncture Resistance. However, Tensile Index did not, being the only tested property that does not correlate with PR. Furthermore, Stretch, TEA and TEA Index are properties that have a good correlation with Puncture Resistance. TEA is the tensile property which better correlates with PR.

Similarly, Khan (2011) concluded that TEA is the paper's property that better correlates with PR. According to the author, this is reasonable once TEA can be defined as the total energy quantity that is absorbed by the material during its straining until its rupture. Therefore, higher TEA value results in higher resistance to puncture [19].

In addition, to better understand these correlations, it is necessary to compare how these tensile properties measurements are similar to Puncture Resistance. As previously mentioned, Puncture Resistance is a function of force and elongation, being calculated as the area below the Force vs. Elongation graph.

The properties with higher correlation with Puncture Resistance, TEA and TEA Index, are also calculated taking in consideration both force and elongation, as the area below the graph, namely U_T (Figure 19). Although it also considers the test pieces' dimensions.

The other properties, with lesser correlation, are only calculated with either force (Tensile Strength) or elongation (Stretch) and the test pieces' dimensions. However, it is clear that Stretch provides higher correlation than Tensile Strength.

7.1.3 Humidity and moisture content

This chapter deals with the influence on the humidity condition and the moisture content of papers in puncture resistance tests.

Two different conditions were used in order to determinate how much a paper's moisture content varies in them and how this change influences on the PR values. The moisture content is show below.

Table 6. *Paper's moisture content comparation in different conditions.*

Paper	Moisture Content		Relative Difference (%)
	50% RH, 23ºC	75% RH, 23ºC	
Flexi	5,71	7,50	31,35
UPM Brilliant Pro	7,62	8,27	8,58
Adv. MG Coating	8,41	10,41	23,87
Adv. Smooth White Strong	6,33	9,54	50,71
PackPro 7.0 Rotogravure	5,40	6,20	14,81
Adv.MG White Cote RB	7,09	7,26	2,42
ClearPack	6,82	7,15	4,84

Table 6 illustrates that moisture content of paper increases while being conditioned in higher relative humidity. This was expected once paper captures moisture from the environment because of its porous cellulose fibers [36].

It is noteworthy that each paper had a difference in absorption, once each paper has a specific composition and papermaking process which can increase or not this phenomenon. Furthermore, the presence of coating/glazed side could impact it as well.

Table 7 illustrates the difference between puncture resistance values while conditioning the paper's in different humidity values.

Table 7. *Paper's Puncture Resistance while in different conditions (Flat Probe).*

Paper	Puncture Resistance		Relative Difference (%)
	50% RH, 23ºC	75% RH, 23ºC	
Adv. Smooth White Strong	4,14	4,94	19,34
Flexi	2,26	2,44	8,15
UPM Brilliant Pro	2,17	2,24	3,06
PackPro 7.0	2,06	2,22	7,72
Adv. MG White Cote	1,89	1,96	3,49
Adv. MG Coating	1,71	2,13	24,49
ClearPack	1,5	1,77	18,14

Paper's puncture resistance increased while being conditioned in an environment with higher humidity. A difference between values were expected because paper's mechanical properties are dependent on its moisture content.

Each mechanical property is differently affected by a paper's moisture content. As previously mentioned, Tensile Energy Absorption tends to increase at increasing amount of moisture contents until a certain quantity [38]. Considering that TEA is the property which better correlates with Puncture Resistance, similar behavior is expected for PR, justifying the increase of its value in the conditioning used.

Comparing Table 6 and Table 7 it is visible that a paper's humidity influences on its puncture resistance, but the relation is not directly proportional. For example, Adv. Smooth White Strong increase its moisture content up to 50% in different conditions, but its puncture resistance did not increase that much.

This phenomenon is better illustrated by the analysis of the following graph (Figure 37) – RH Difference (%) vs. PR Difference (%) – which resulted in R^2 nearly to zero. It depicts that when a paper is accommodated in different conditions, the change of relative humidity caused by those conditions do not correlate with the variation in puncture resistance values.

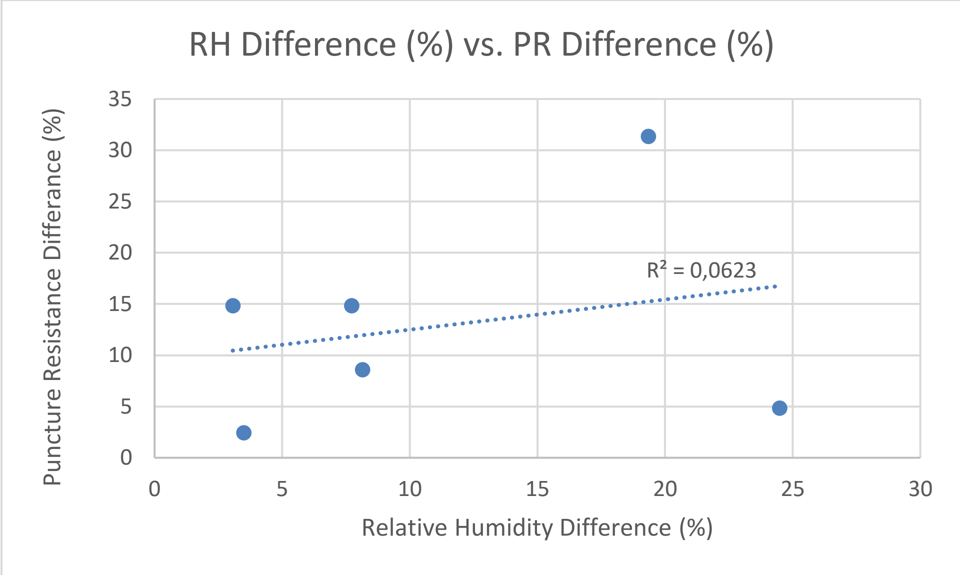


Figure 37. *RH Difference (%) vs. PR Difference (%).*

7.1.4 Paper grammage

Paper grammage will be analyzed in this section. Initially, it will be discussed how puncture resistance varies when comparing the same paper type, but with different grammages. Later on, it will be studied puncture resistance of different papers with the same grammage.

In order to analyze how a paper's grammage influences on its puncture resistance, the same paper type, but with different grammages were analyzed. The following table depicts the Puncture Resistance values of Advantage Formable with two different grammages.

Table 8. *Puncture Resistance of Adv. Formable with different grammages.*

Adv. Formable Puncture Resistance (mJ)	Grammage				Relative Difference (%)
	100 g/m²	σ	130 g/m²	σ	
Flat Probe	9,77	1,05	12,85	1,52	31,49
0,8mm Hemis. Probe	5,84	0,80	8,02	1,02	37,28

Table 8 illustrates that a specific paper's puncture resistance is directly proportional to its grammage. The increase of 30% in grammage between papers, resulted in an increase of around 30% in puncture resistance using different probes. A higher relative difference can be justified by standard deviation in PR values.

The following graph illustrates the Puncture Resistance (50% RH) of different paper types of same grammage. Both ClearPack and Flexi papers have grammage of 62 g/m^2.

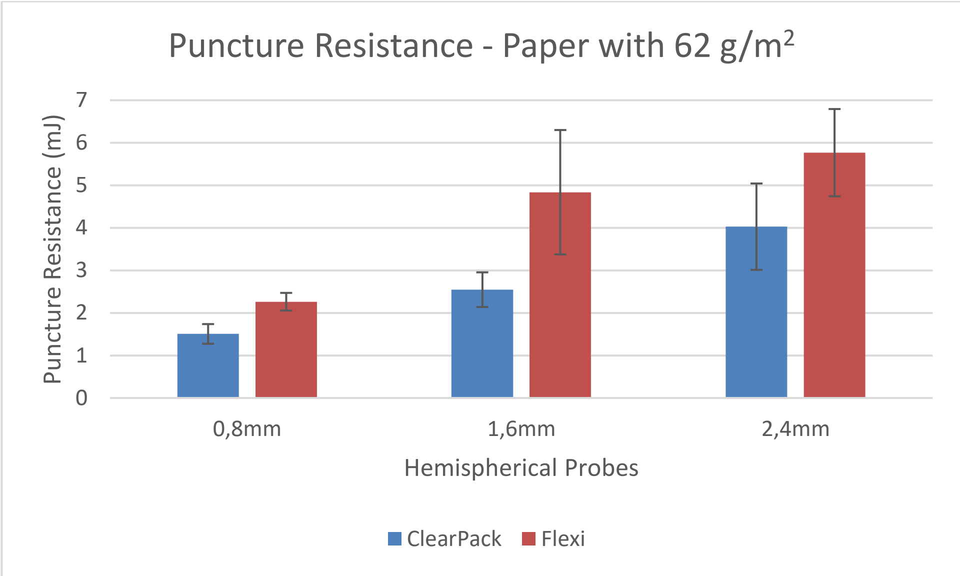


Figure 38. *Puncture Resistance of different paper with same grammage.*

Figure 38 illustrates that even though ClearPack and Flexi have the same grammage, their puncture resistance values are different. Therefore, grammage do not correlate with puncture resistance, while different papers types are being considered.

A specific paper type will have its puncture resistance proportional to its grammage. However, this is not true while comparing different types of papers with similar grammage.

7.1.5 Presence of coating/glazed side

The presence of coating and glaze side and their influence on puncture resistance will be analyzed in this section. The following figure illustrates the PR's values of HS Bag Wgon, in both sides – normal (uncoated) and coated.

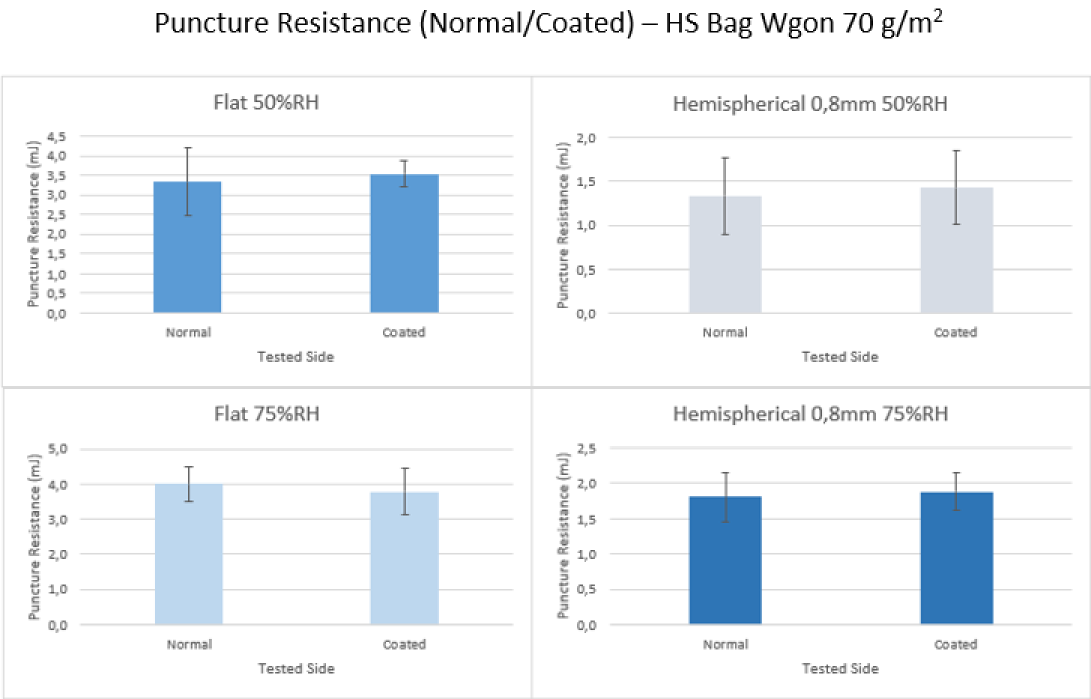


Figure 39. *Comparation of Puncture Resistance of Normal and Coated side.*

Figure 39 illustrates the comparation of PR values while testing this paper both sides: uncoated and coated. Regardless of the paper's conditions or probe used, its puncture resistance is not influenced by which side it is measured, thus the presence of coating did not change the PR's values. The same behavior was presented in all the coated papers tested in this thesis, with all variables used.

The following figure illustrates PR's values of Adv.MG Kraft, in both sides – normal and glazed.

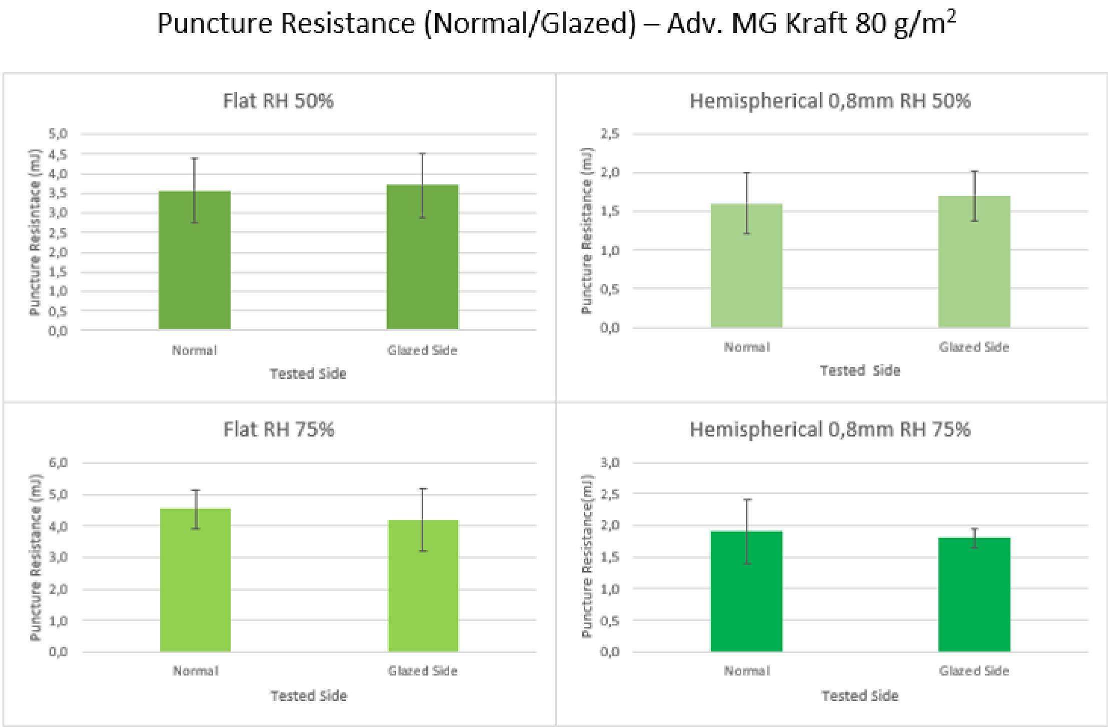


Figure 40. *Comparation of Puncture Resistance of Normal and Glazed side.*

Figure 40 depicts the comparation of PR values while testing this paper both sides: normal and glazed. Regardless of the paper's conditions or probe used, its puncture resistance is not influenced by which side it is measured, thus the presence of the glaze did not change the PR's values. The same behavior was presented in all the glazed papers (MG papers) tested in this thesis, with all variables used.

For the paper's types tested in this thesis, the presence of coating/glazed sided did not influence on a paper's puncture resistance. Considering that their quantity is very limited, it did not change a paper's mechanical strength.

7.1.6 Bleaching influence

This section deals with the influence of a pulp bleaching in puncture resistance of paper. Table 9 illustrates the puncture resistance crescent values (Flat Probe), of bleached and unbleached papers.

Table 9. *Puncture Resistance (Flat Probe) of Unbleached & Bleached papers.*

Paper	PR (mJ)	σ	Bleached/ Unbleached
ClearPack	1,51	0,23	Bleached
Adv. MG Coating	1,71	0,26	Unbleached
Adv. MG White Cote RB	1,89	0,26	Bleached
PackPro 7.0	2,07	0,39	Bleached
UPM Brilliant Pro	2,17	0,35	Bleached
Flexi	2,26	0,21	Bleached
BAG WGOM 70	2,57	0,68	Bleached
HS BAG Wgom	3,33	0,88	Bleached
Adv. MG Kraft 80	3,57	0,81	Unbleached
Adv. Smooth W. Strong	4,14	0,74	Bleached
Adv. MF Z	5,24	0,74	Unbleached
Adv. Formable 100	9,77	1,05	Unbleached

Table 9 illustrates that unbleached paper tends to have higher puncture resistance. This is expected because, in general unbleached paper is stronger than bleached one [30]. However, this is not always true as illustrated by Adv. MG Coating paper, which regardless of being bleached provided one of the lowest PR values of the papers analyzed.

Therefore, if the bleaching process influences on a paper's final strength, it will consequently affect its puncture resistance. Although some papers need this process to be used in packaging, it is necessary to pay attention to how bleaching would affect its strength.

7.1.7 Paper materials

The paper materials (pulps) are further examined in this section. First, it will be selected papers made of the same pulp material in order to check their puncture resistance values. Table 10 illustrates puncture resistance of papers made of pure bleached long-fiber sulphate pulp.

Table 10. *Puncture Resistance (Flat Probe) papers made of pure bleached long-fiber sulphate pulp.*

Paper	PR (mJ)	σ
Adv. MG Coating	1,71	0,26
Adv. MG Kraft 80	3,57	0,81
Adv. Formable 100	9,77	1,05

Table 10 depicts that papers made of the same material can have very different puncture resistance values. Therefore, not only the pulp used but also its papermaking process influences on a paper puncture resistance.

Furthermore, it can also be studied papers made of materials combination. This is the case of Adv. Smooth White, which is made of bleached long-fiber sulphate pulp and bleached short-fiber sulphate pulp. As seen on Table 9, this paper is one of the strongest of all tested in this thesis, despite being a bleached paper.

According to Schennach et al. (2015) a paper's strength results from its fiber's strength and fibers bonding strength [57]. The combination of both fibers used, its strength and degree of bonding, could then justify the high strength of Adv. Smooth White.

It can be concluded that a paper's puncture resistance is influenced by its material. Also, that the combination of materials used in a paper can increase its puncture resistance, which can be interesting in cases where stronger bleached papers are required.

7.2 Paper's puncture resistance and driving factors

The final objective of this research project is to analyze what are the driving factors that contribute for a paper's puncture resistance. This thesis studied different papers and their puncture resistance in different conditions. This section deals with a few reasons to justify the difference in this property between tested papers.

It was already mentioned that the pulp's quality and how the papermaking is performed shapes a paper's strength. Therefore, one key driving factor is the pulp used. The others crucial factors are the addition of fillers and the refining process, which is a part of the paper production.

It was verified that Tensile Energy Absorption (TEA) is the tensile property which betters correlates with Puncture Resistance. According to this correlation, both TEA and Puncture Resistance would have similar driving factors. During papermaking, the addition of fillers and the refining process influences on a paper's Tensile Energy Absorption.

As previously mentioned, the addition of fillers in paper is commonly done to reduce its production costs. However, this addition reduces the amount of fibers, which consequently reduces the paper's strength. As seen on Figure 8, TEA Index is strongly reduced by the filler content, thus this will also drastically reduce a paper's puncture resistance.

It is important to take in consideration that the addition of filler will not only reduce a paper's production costs, but it will also decrease its puncture resistance. Therefore, for papers used as packaging materials, the amount of fillers should be reduced in order to produce a paper with sufficient puncture resistance.

Lastly, the influence of the refining process will be analyzed. According to Johansson (2011) each pulp will behave differently to the refining process and this stage should be adjusted by the fiber's modifications needed for a specific paper. This process can shorten the fibers, which improves the paper's formation. Also, the beating improves fibers' surface area and flexibility. Flexible fibers conform around each other, once they are more bendable [58].

Furthermore, during this process fines are produced. The combination of fines and fiber flexibility increases the contact between fibers, which improves fiber joints strength, thus the paper's strength [58].

According to Bäckström et al. (2008), fines have a high influence on the paper's final mechanical properties. There are two types of fine: primary fines, which are already present in the pulp before the refining process; and secondary fines, which are created during this process. The addition of fines in paper, particularly the secondary type, improves its Tensile Index, Burst Index and TEA [59].

The driving factors for a paper's Puncture Resistance would also be the factors which affect Tensile Energy Absorption, considering their correlation. Therefore, the pulp used, the filler content, and the refining process influence on a paper's Puncture Resistance.

Finally, to produce a paper with high puncture resistance it is necessary the correct selection of the pulp and its refining process that consequently results in a paper with good strength. Furthermore, the amount of filler should be reduced.

8. CONCLUSIONS

This thesis analyzed Puncture Resistance of papers used as sustainable packaging materials. Puncture Resistance is a material property which defines how much a material can withstand external piercing forces.

This property is essential in package material because a packaging made of high puncture resistance material will be stronger against any eventual damage a package can suffer. This collaborates for the safety and quality of the product packaged.

Different probes were used during this research project, in order to emulate the impacts a paper can suffer in real life conditions and to analyze how strong they are against then. In total, 5 different probes were manufactured and tested. Also, the tools required for the experiments were designed and produced.

Stress concentration is the key element which justifies the differences in a paper's puncture resistances while using the probes. Higher stress concentration in paper during the process, facilitates it to be pierced, which decreases its puncture resistance.

Flat probe proved to causes less stress concentration than hemispherical one, thus provides greater puncture resistance. Also, while comparing probes with same tip geometry, puncture resistance will present direct proportion with its tip dimension. Finally, same format probes, but with different tip materials (hard and soft), did not cause significant change in a paper's puncture resistance.

It was proven that Tensile Energy Absorption is the tensile property which best correlates with Puncture Resistance. From this correlation it was viable to determinate that paper's filler content and the refining process are possible driving factors that influence on the paper's puncture resistance. Also, the pulp used was considered another factor that collaborates for this property because this dictates the sheet's final strength.

It was concluded that to produce a paper with high puncture resistance, it is necessary to correctly select a pulp and its refining process. Also, the amount of fillers used should be reduced.

Furthermore, paper grammage, the presence of a coating/glazed side, bleaching, paper materials and moisture content were studied to check their relevance on a paper's puncture resistance. It was demonstrated that a specific paper type will have its puncture resistance directly proportional to its grammage. For the paper's types tested in this research project, the presence of coating/glazed side did not prove to affect a paper's puncture resistance.

It was analyzed that in overall unbleached pulps will provide higher PR than bleached ones. Also, it was concluded that different papers made of the same pulp type can have different puncture resistance values depending on its papermaking process and that a paper made of combinations of materials, can be stronger.

Finally, the increase of paper's moisture content improved its puncture resistance, which was expected once some paper's mechanical properties improve with a higher moisture content. However, this relation is not directly proportional.

As recommendations for further work would be interesting to analyze how different relative humidifies would affect a paper's puncture resistance. Also, to study how puncture resistance values would be modified by different soft probes and perforation angles.

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APPENDIX

Table 11. *Paper's Tensile Strength.*

Paper	Tensile Strength (Kn/m)	σ	Tensile Strength (Kn/m)	σ	Geometric mean (Kn/m)
	MD	**MD**	**CD**	**CD**	
UPM Brilliant P.	6,01	1,33	2,96	0,15	4,22
HS BAG Wgom	5,85	0,62	3,31	0,23	4,40
BAG WGOM 70	4,88	1,04	2,99	0,21	3,82
Adv. MG White Cote RB	3,19	0,82	2,48	0,31	2,81
Adv. MG Coating	2,62	0,61	1,74	0,48	2,14
Adv. MF Z	4,54	0,33	2,42	0,19	3,31
Adv. MG Kraft (80g/m^2)	6,24	1,40	4,54	0,39	5,32
Adv. Formable (100g/m^2)	7,59	0,50	5,90	0,67	6,69
Adv. Smooth White S.	8,01	1,20	4,15	0,12	5,77
PackPro 7.0 Roto.	4,51	0,28	2,28	0,05	3,21
ClearPack	5,62	0,63	2,52	0,41	3,76
Flexi	2,19	0,02	4,62	1,05	3,18

Table 12. *Paper's Tensile Index.*

Paper	Tensile Index (Nm/g)	σ	Tensile Index (Nm/g)	σ	Geometric mean (Nm/g)
	MD	**MD**	**CD**	**CD**	
UPM Brilliant P.	96,99	1,33	47,74	0,15	68,05
HS BAG Wgom	66,43	0,62	37,63	0,23	50,00
BAG WGOM 70	69,66	1,04	42,77	0,21	54,58
Adv. MG White Cote RB	67,80	0,82	52,84	0,31	59,85
Adv. MG Coating	65,49	0,61	43,52	0,48	53,39
Adv. MF Z	90,84	0,33	48,31	0,19	66,24
Adv. MG Kraft (80g/m^2)	77,96	1,40	56,70	0,39	66,49
Adv. Formable (100g/m^2)	75,87	0,50	59,02	0,67	66,92
Adv. Smooth White S.	100,11	1,20	51,88	0,12	72,07
PackPro 7.0 Roto.	64,40	0,28	32,56	0,05	45,79
ClearPack	90,62	0,63	40,60	0,41	60,66
Flexi	35,39	0,02	74,49	1,05	51,35

Table 13. *Paper's Stretch.*

Paper	Stretch (%)	σ	Stretch (%)	σ	Geometric mean (%)
	MD	MD	CD	CD	
UPM Brilliant P.	2,09	0,46	6,68	0,75	3,73
HS BAG Wgom	1,67	0,00	7,52	1,40	3,54
BAG WGOM 70	1,81	0,34	7,65	1,11	3,72
Adv. MG White Cote RB	1,25	0,46	2,78	0,86	1,87
Adv. MG Coating	1,17	0,46	1,67	0,59	1,40
Adv. MF Z	6,85	0,70	5,57	1,36	6,17
Adv. MG Kraft (80g/m^2)	1,81	0,34	3,06	0,43	2,35
Adv. Formable (100g/m^2)	10,19	0,70	9,60	1,08	9,89
Adv. Smooth White S.	2,09	0,46	6,12	1,14	3,58
PackPro 7.0 Roto.	1,88	0,42	7,52	0,27	3,76
ClearPack	2,37	0,34	1,50	0,37	1,89
Flexi	2,92	0,59	2,23	0,86	2,55

Table 14. *Paper's TEA.*

Paper	TEA (J/m^2)	σ	TEA (J/m^2)	σ	Geometric mean (J/m^2)
	MD	MD	CD	CD	
UPM Brilliant P.	53,67	26,18	137,53	0,75	85,92
HS BAG Wgom	58,32	9,19	189,67	1,40	105,17
BAG WGOM 70	50,32	19,34	168,39	1,11	92,05
Adv. MG White Cote RB	21,58	9,62	50,39	0,86	32,98
Adv. MG Coating	11,74	6,87	20,26	0,59	15,42
Adv. MF Z	193,66	35,54	99,23	1,36	138,63
Adv. MG Kraft (80g/m^2)	44,29	17,79	86,85	0,43	62,02
Adv. Formable (100g/m^2)	482,35	31,09	351,85	1,08	411,96
Adv. Smooth White S.	116,46	36,90	169,78	1,14	140,62
PackPro 7.0 Roto.	47,57	11,69	130,79	0,27	78,87
ClearPack	82,05	20,01	20,11	0,37	40,62
Flexi	43,45	16,47	66,60	0,86	53,80

Table 15. *Paper's TEA Index.*

Paper	TEA Index (J/kg)	σ	TEA Index (J/kg)	σ	Geometric mean (J/kg)
	MD	**MD**	**CD**	**CD**	
UPM Brilliant P.	865,72	26,18	2218,16	23,11	1385,75
HS BAG Wgom	662,67	9,19	2155,28	48,69	1195,09
BAG WGOM 70	718,80	19,34	2405,52	33,31	1314,94
Adv. MG White Cote RB	459,19	9,62	1072,14	23,48	701,66
Adv. MG Coating	293,39	6,87	506,49	11,20	385,49
Adv. MF Z	3873,20	35,54	1984,70	30,87	2772,57
Adv. MG Kraft (80g/m^2)	553,61	17,79	1085,64	17,18	775,26
Adv. Formable (100g/m^2)	4823,46	31,09	3518,52	134,88	4119,64
Adv. Smooth White S.	1455,77	36,90	2122,26	44,94	1757,71
PackPro 7.0 Roto.	679,54	11,69	1868,38	15,17	1126,78
ClearPack	1323,33	20,01	324,32	7,79	655,12
Flexi	700,87	16,47	1074,18	27,65	867,67