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IMPACT OF PREPARATION PROCEDURE ON LAB RESULTS FOR RUBBERIZED REINFORCEMENT MATERIALS

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Abstract

Nowadays, the modern tire is designed to satisfy specific requirements. Textile reinforcement plays a crucial role in improving the structural integrity and overall performance of tires. Reinforcement testing methods are employed to evaluate the effects of different textile materials on tire performance parameters. Prior to testing, proper preparation of materials is essential.

This work studies the impact of different preparations procedure on lab results for rubberized reinforcement materials with the aim of developing an optimal procedure that can guarantee reliable and reproducible results in all laboratories. Therefore, force-elongation behaviors and twist level values were investigated. An additional analysis was done by a coordinated cross-check testing between different laboratories to verify the comparability of a test performed in multiple laboratories.

Through observation, it was evident that cap strip materials displayed a preference for specific preparation methods, while calendered materials exhibited a preference for different preparation procedures. As for the conditioning, the methods tested did not affect the force-elongation curves and, in general, laying with a knot (L/N) presented more reliable results.

In the cross-check testing, it could be observed that even materials coming from the same production process, the handling, conditioning, and testing have an impact onto the results. To comprehend the results and reach meaningful conclusions, it is necessary to replicate the tests.

In summary, the preparation procedure is advisable to be done with clean, dry hands, with the possibility of using rubber gloves. As for the cord-pulling position, it is recommended to do it randomly (RP) - considering that in this preparation the cord chosen to pull out would come out easily and without much effort and tension. For cap strip materials, pulling only the rubber on the edges can facilitate the extraction. For calendered materials, pull the cord from the edge of the strip and pulling only the cord out of rubber compound also contribute for an easier the procedure.

Keywords (theme): Textile reinforcements; samples preparation; tire reinforcement testing; cross-check.

Resumo

Atualmente, o pneu moderno é projetado para satisfazer requisitos específicos. O reforço têxtil desempenha um papel crucial na melhoria da integridade estrutural e do desempenho geral dos pneus. Testes em reforços são empregados para avaliar os efeitos de diferentes materiais têxteis nos parâmetros de desempenho do pneu. Antes dos testes, a preparação adequada dos materiais é essencial.

Este trabalho estuda o impacto de diferentes procedimentos de preparação nos resultados laboratoriais de materiais de reforço de borracha, com o objetivo de desenvolver um procedimento ideal que possa garantir resultados confiáveis e reprodutíveis em todos os laboratórios. Portanto, os comportamentos de força-elongação e os valores de torção (*twist*) foram investigados. Uma análise adicional foi realizada por meio de testes coordenados para verificar a comparabilidade de um teste realizado em vários laboratórios.

Por meio da observação ficou evidente que os materiais de *cap strip* exibiam preferência por métodos de preparação específicos, enquanto os materiais calandrados apresentavam preferência por diferentes procedimentos de preparação. Quanto ao condicionamento, os métodos testados não afetaram as curvas de força-elongação e, de forma geral, o condicionamento deitado com um nó (L/N) apresentou resultados mais confiáveis.

Nos testes coordenador de verificação, foi possível observar que mesmo materiais provenientes do mesmo processo de produção, o manuseio, o condicionamento e os testes têm um impacto nos resultados. Para compreender melhor os resultados e chegar a conclusões significativas, é necessário replicar os testes.

Em resumo, o procedimento de preparação é aconselhável ser realizado com mãos limpas e secas, com a possibilidade de usar luvas de borracha. Quanto à posição de puxar a corda, é recomendado fazê-lo aleatoriamente (RP) - considerando que, nessa preparação, a corda escolhida para ser retirada sairia facilmente e sem muito esforço e tensão. Para materiais de *cap strip*, puxar apenas a borracha nas bordas pode facilitar a extração. Para materiais calandrados, puxar a corda a partir da borda da faixa e puxar apenas a corda fora da borracha também contribui para facilitar o procedimento.

Palavras-Chave (Tema): Reforços têxteis; preparação de amostras; teste de reforços de pneu; *cross-check*.

Declaration

I hereby declare, under word of honor, that this work is original and that all non-original contributions is indicated, and due reference is given to the author and source

3rd July 2023

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Notation and Glossary

EASL	Elongation At Specific Load	%
LASE	Load At Specific Elongation	N

List of Acronyms

AG	Aktiengesellschaft
ASTM	American Society for Testing and Materials
EPDM	Ends of cord per dm width of fabric
FEUP	Faculty of Engineering of Porto
ISO	International Organization for Standardization
PA	Polyamide
PET	Polyethylene terephthalat
SEC	Single end cords

1 Introduction

The present dissertation was developed under the guidance of Professor Fernão Magalhães as part of the requirements for a master's degree in chemical engineering conferred by Faculty of Engineering of University of Porto (FEUP). The research work was accomplished in collaboration with Continental Aktiengesellschaft (AG) at the facility for Body Compound and Reinforcement Technology and it took place from February to June 2023.

1.1 Framing and presentation of the work

Tires originally began as wheels, one of the most important inventions in human history, because of their wide applications such as vehicles, construction equipment, and internal parts of machinery [1, 2]. It started at least 4000 years ago with round slices of logs. These were followed by discs made of wood joined together and then by spoked wheels, with spokes encircled by a rim and, first a leather band then, an iron band around the outside to prevent damage. Despite that iron or wooden wheels had difficulties functioning on rough road surfaces and did not offer comfort riding [2].

Nowadays, the modern tire is designed to satisfy specific requirements and it is composed of rubber, fillers, reinforcements, plasticizers, and other chemicals in different quantities for each type of tire. A different composition of material provides different properties [3]. The proportion of material in a Continental's summer model is [4]:

- Rubber 41%;
- Fillers 30%;
- Reinforcing materials 15%;
- Plasticizers 6%;
- Chemicals for vulcanization 6% and;
- Anti-ageing agents and other chemicals 2%.

Notwithstanding the fact that there are tires with different designs and applications, all of them must fulfill a set of functions [5]:

1. Provide load-carrying capacity.
2. Provide cushioning and dampening.
3. Transmit driving and braking torque.
4. Provide cornering force.
5. Provide dimensional stability.
6. Resist abrasion.
7. Generate steering response.

8. Exhibit low rolling resistance.
9. Provide minimum noise and minimum vibration.

Given attention to the reinforcements, they contribute to most of the functions required. Tire reinforcement materials are the predominant load-carrying members of the cord-rubber composite providing a stable motor vehicle performance over the needed operating conditions as well as keeping the air pressure inside and giving strength to the other tire parts such as the tread and sidewall [5-7].

The reinforcement materials are made of textile and steel. In textile reinforcements, the individual filaments are twisted into a yarn and then two or three yarns are twisted together to form a cord. The cords are dipped in a coating that provides chemical bonding that will be further activated by the heat and curing process followed by a high-tension treatment. The reinforcement is present in the tire in the form of cords – single end cords (SEC) or fabrics. In the second one, the cords are crossed by threads, so that the cords keep their spacing between them, however, these threads are destroyed during the tire building process. Then, the material can pass through either two rubberizing processes: calender, for fabric material or cap strip machines, for SEC. In the first one, the textile cords are coated with a thin layer of rubber on both sides (topping), and in the second, the cords are impregnated with rubber forming a strip that is later wound up [2, 6, 8]. After these processes, the material is cooled down and sent to the quality test laboratory.

When it comes to reinforcement testing, it is mainly performed for the verification of the reinforcement requirements as well as reinforcement comparison before and after rubberizing it, confirming if the material maintains its properties even after all those procedures. The dipped material (non-rubberized) is used as a reference and the rubberized material is compared to that.

Since during the manufacturing process, the textile reinforcement faces temperature, tension, and moisture changes, the material is stored and transported like this and it does not arrive at the laboratory in a steady state, instead, it arrives in a state affected by those parameters. In order to have repeatable and stable test results, the material is previously conditioned in a standard laboratory climate and relaxed storage, which implies that any tension that may exist when the material arrives can be relaxed and reach a steady state making repeatable and comparable results possible.

However, rubberized, and non-rubberized materials have different preparations. The dipped material comes in a spool, as a SEC, or a fabric, which makes easier the separation of the cord to conditioning. Meanwhile, to take the cord out of the rubber matrix is necessary to apply a tension that is not controlled.

The preparation impacts might be contributing to deviations in the results. On account of this, this work aims to study the impact of those procedures on the tire rubberized cord, especially the impact of the preparation reflected in the quality test results. Several parameters of sample conditioning and preparation were studied in order to accomplish an optimal procedure that can guarantee reliable and reproducible results in all laboratories.

1.2 Presentation of the company

Continental (former Continental Caoutchouc & Gutta-Percha) company was founded in Hanover in 1871, fifteen years before the manufacturing of the first automobile. Producing rubberized fabrics for raincoats, hot-water bottles, and solid tires for bicycles and carriages, in 1892, it became the first German company to manufacture pneumatic tires for bicycles. However, it was at the beginning of the 20^{th} century that the company turned its focus to automobile tires [9].

Nowadays, Continental is the pioneer in developing technologies and services for sustainable and connected mobility of people and their goods, offering safe, efficient, intelligent, and affordable solutions for the automobile industry. The company is present in almost 60 countries and markets and in 2022 it generated sales of 39.4 billion euros [9].

Also, the company underscores its own sight of becoming the most progressive tire company in the world and it is committed to leading the way in terms of environmental and social responsibility by the year 2030 [9].

Since 2022, the Continental Group has been divided into three business areas [9]:

Tires: passenger car, light truck, commercial vehicles, two wheels and specialty (agricultural, mining, etc.) tires.

Automotive: architecture and networking, autonomous mobility, safety and motion, smart mobility, and user experience.

ContiTech: advanced dynamics solutions, conveying solutions, industrial fluid solutions, mobile fluid systems, power transmission group, surface solutions.

1.3 Contribution of the author to the work

This work took as its first approach the review and analysis of the current procedures for preparing and conditioning textile cords, identifying the possible results impact factors. Several different preparations were tested with respect to handling, the position and means of removing the cord from the rubber, and the speed with which to do so. About conditioning, different conditions were tested to find the method that best preserved the characteristics of the materials, especially the twist level. As for the cross-check testing, besides the tests performed by the author, other 4 laboratories provided data from their tests, in other to compare the results. With the results and practical experience gained, an optimization of the existing procedure was developed in a clear way, ensuring reliability and repeatability.

1.4 Organization of the dissertation

This dissertation is divided into 7 chapters. The brief presentation of each one of these chapters is described as follows:

1. Introduction: An overview of the studied topic and the aim of the study as well as the presentation of the company.
2. Context and State of the Art: Provides background information required for the understanding of the study performed. This includes bibliographic research on the key concepts of tire technology, textile reinforcements properties, and manufacturing procedure.
3. Materials and Methods: A description of the materials and equipment used along with the test procedures.
4. Discussion: Presents a detailed analysis of the study performed.
5. Conclusions: A brief description of the outcome of the results is presented.
6. Assessment of the work done: It describes the degree of accomplishment of the objectives and the limitations of the main work.
7. References: A list of all documents searched through the writing of this dissertation.

Appendices: Contain additional and supportive information.

2 Context and State of the art

This chapter presents an overview of the key concepts of tire technology, especially textile reinforcements properties and manufacturing procedures. To support the research, a bibliography review of these topics is provided.

2.1 Tires summary

In today's global market, the tire can be found in two types of construction, either a bias ply construction or a radial Figure 1. In bias tires, the fabric plies are overlapped in a diagonal configuration extending from bead to bead, resulting in a composite network of layers running in opposing directions [2, 5].

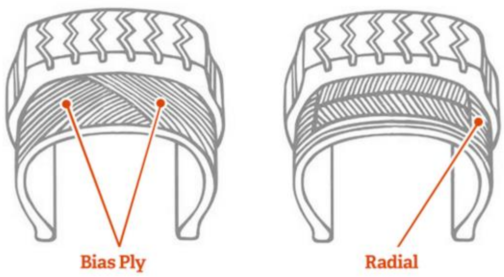


Figure 1. Demonstration of bias ply and radial tire (Adapted from [10]).

The radial tires, invented in the 1950s, have widely replaced the bias tires on the market; they have a less rigid casing construction, where flexible ply cords extend transversely or radially from one bead to the other bead. The reason why radial tire became so popular is that it fits the user's needs in terms of stability and controllability at high speeds, as well as better wear resistance and lower fuel consumption at high speeds.

Tire components can be grouped into two main elements, the tread and belt assembly and the casing, consisting of several layers that can be described as follows for a passenger car tire Figure 2:

Figure 2. Passenger car tire design [11].

1. Tread: rubber component of the tire that it is in contact with the road, and it must guarantee the necessary traction for driving and braking. Its compound is specially formulated to provide a balance between wear, traction, handling, and rolling resistance [5, 6]. Underneath the tread, there is the tread base which contributes to good adhesion between the tread and the tire casing, as well as reducing the damage to the casing [11].
2. Jointless cap plies: directly below the tread, it enables travel at high speeds. They consist of small strips of rubber with textile reinforcement applied around the circumference of the tire, from one side to the other, without overlapping [3, 5, 11].
3. Steel cord belt plies: steel cords that provide rigidity to the tire. This layer provides improved wear performance, vehicle stability, handling performance, damage resistance, and protection of the ply cords [5, 11].
4. Textile cord ply: extended fabric layer from the bead on one side to the opposite side. It gives the strength that allows the tire to retain air at high pressures, also controls its internal pressure, and maintains its shape [11].
5. Inner liner: a membrane consisting of compounded low permeability rubber, such as butyl rubber, which the main function is to retain the compressed air inside the tire. It seals the air-filled inner chamber, from the bead on one side of the tire to the opposite side [5, 11].
6. Sidewall: tire sidewall rubber protects the casing from abrasion, impact, and flex fatigue. Its compound is formulated to resist cracking due to environmental hazards such as ozone, oxygen, UV radiation, and heat. Provides long-term protection and casing durability [3, 5, 6, 11].
7. Bead reinforcement: it is in the region that helps the tire to be sealed to the wheel (rim) thanks to air pressure (bead). The reinforcement is a strong, heat-resistant, synthetic fiber. It improves direction stability and facilitates steering response [5, 11].
8. Bead apex: made of a compound formulated for dynamic stiffness. It performs the same tasks as the bead reinforcement and provides additional steering comfort [5, 11].
9. Bead core: a steel wire impregnated in a rubber layer that ensures that the tire is placed firmly on the rim [5, 11].

2.2 Textile Reinforcements

As previously mentioned, textile cords provide the reinforcement necessary for the tire to maintain its dimensions while under external and internal factors and to carry the load at the vehicle-operating speed [3, 5]. The tire reinforcement science implements a specialized terminology that is important to be familiar with while studying it. This terminology is described in Appendix A -.

2.2.1 Textile materials

Taking into consideration the performance demands of fatigue resistance, structural rigidity, tensile strength, durability, and resilience, a range of polymer-based cords are suitable for tire application, such as rayon, nylon, polyester, and aramid [3, 5].

Rayon was the first synthetic fiber used as tire reinforcement, made from cellulose. It can be drawn or stretched up to 100% of its original length to that enable crystalline orientation to produce a high-tenacity rayon suitable for tires. The main features of rayon are high tensile strength, high dimensional stability, and good fatigue and heat resistance. Its limitations involve its high sensitivity to moisture, its price, and the environmental manufacturing issues regarding Rayon's facilities. In moist conditions, their dry strength is significantly reduced, as a result, the processes that involve rayon must have a strictly controlled humidity [1, 2, 5, 6].

Nylon is a polyamide (PA) polymer characterized by the presence of amide groups in the main polymer chain. Despite the large variety of nylon available, only two are being applied in tire technology: nylon 6.6 and nylon 6. Both types contain the same chemical groups in the same proportion which implies very similar chemical properties. Still, there exists a subtle difference in the arrangement of the molecules that result in slightly lower crystallinity values for nylon 6 compared to nylon 6.6. The key properties of this material are its excellent tensile strength, heat, and abrasion resistance, good elastic recovery, and less sensitivity to moisture compared to rayon. Its constraints are related to its thermal shrinkage. Since nylon is a thermal plastic, when heated it tends to shrink. Because of that, it requires a process of cooling while still inflated after vulcanization. The morphological structure of high-strength nylon consists of microfibrillar and inter-microfibrillar regions (Figure 3). The first one consists of crystalline and amorphous regions, as the second one, oriented molecular chains [2, 5, 6, 12, 13].

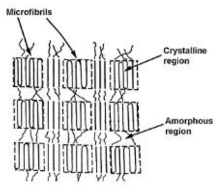


Figure 3. Microstructure of highly drawn PA fibers ([13]).

Polyester is a polymer containing ester groups in its main polymeric chain. Polyester is usually far more stable and less reactive than simple aliphatic esters. Like nylon, a range of polyester are available commercially for use in tires but the most used is polyethylene terephthalate (PET). PET is mostly used for having low shrinkage and moisture sensitivity, good fatigue and abrasion resistance, and high tensile strength as well as a low cost. Polyester is a combination of the strength and elongation characteristics of the nylons with the firmness characteristics of the rayons. Despite that, the polyester cord is not recommended for use in high-load/high-speed/high-temperature applications, because it easily loses properties at tire temperatures above about 120 ºC. The polyesters are composed of a partially amorphous and partially crystalline structure (Figure 4) [5, 6, 12, 13].

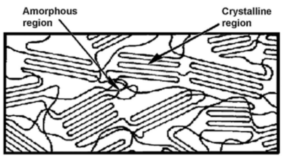


Figure 4. Microstructure of polyester fiber [13].

Aramid is another type of fiber that is applied to tires. 'Aramid' is short for aromatic polyamide, meaning it belongs to the same class as nylons. It has twice the strength per weight as nylon, being comparable to steel strength, and around eight times the elastic modulus. In addition, aramid has excellent resistance to heat, chemicals, and abrasion. The disadvantage of using aramid is regarded its expensive price, the difficulty of adhesion to rubber, and a low value for elongation at break, which can cause difficulty in some applications. The structure of aramid fiber consists of highly orientated and extended chains, packed together with a very high degree of crystallinity (Figure 5) [2, 5, 12, 13].

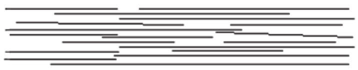


Figure 5. Microstructure of aramid fiber [13].

Nowadays, in addition to the use of cords made of only one material with properties as described above, hybrids cords have been developed. They have stood out for being new reinforcement materials with better performance using conventional fibers. A hybrid cord is a combination of at least two different materials, twisted together improving their own properties by compensating for the conflicting needs for strength, fatigue resistance, and adhesion [5, 14].

On top of that, hybrid cords offer incomparable properties with economic advantages to meet the increasing demands of the tire industry. Several patents present their preparation and applications [15]. The most common combinations are polyester with nylon and aramid with nylon.

The main characteristics of the textile fibers presented are summarized in Table 1.

Table 1. Properties of major textile tire reinforcements (adapted from [7, 12, 16]).

Material properties	Rayon	Nylon	Polyester	Aramid
Moisture regain / %	13.0	4.5	0.5	2.5
Heat resistance / ºC	150.0	180.0	180.0	250.0
Glass transition temperature / ºC	-	40.0 – 55.0	80.0	-

Material properties	Rayon	Nylon	Polyester	Aramid
Melting point / °C	Decomposes above 210.0	Nylon 6 – 225.0 Nylon 6.6 – 250.0	250.0	Decomposes above 500.0
Tenacity / $cN \cdot Tex^{-1}$	50.0	Nylon 6 - 80.0 Nylon 6.6 - 85.0	80.0	190.0
Elongation at break / %	6.0	Nylon 6 - 19.0 Nylon 6.6 - 16.0	13.0	4.0
Initial modulus / $cN \cdot Tex^{-1}$	800.0	Nylon 6 - 300.0 Nylon 6.6 - 500.0	850.0	4000.0
Shrinkage at 150°C / %	0	Nylon 6 - 6.0 Nylon 6.6 - 5.0	2.0*	0.2

*Polyester applied in tire industry is HMLW (high modulus, low shrinkage)

Each type of tire contains at least one type of these fibers in its structure depending on the specification of that tire cord. Table 2 presents the different types of tires and the most used material.

Table 2. Example of type of fiber used in different tires (Adapted from [7]).

Tire type	Tire cord
Racing	Rayon
Truck Heavy duty Aircraft	Nylon
Radial passenger and light truck	Polyester
Radial aircraft	Aramid

In order to obtain the finest benefit from the application of fibers as reinforcement in rubber compounds, it is necessary to modify them from filaments into cords. The filaments are twisted into a yarn and the yarns, twisted again, and processed into cords. If the cords are going to become a fabric, they are woven with pick cords to maintain spacing, into a wide sheet. After the cord or fabric processing, the material is dipped in a latex coating to enhance bonding to rubber followed by a high-tension and heat treatment. The material is then passed through a calender, to change into a rubber sheet, or to a cap strip line, to produce a cap-ply [5, 6].

2.2.2 Cord construction

The process of cord construction starts with the production of continuous filaments from the polymers, which can be done by melt spinning, for nylon and polyester fibers, and solution spinning, for rayon and aramid [6, 13].

In the melt spinning (Figure 6), the molten polymer is filtered and pumped through a creel with a big number of very thin holes. The extruded semi-molten polymer is subjected to a drawing process to improve the fiber properties, where it is stretched to about 25 times its original length while solidifying in a cool air stream. This procedure occurs while the polymer is still above its glass transition temperature, and it contributes to the increase of the polymer's strength and modulus as well as to the reduction of its breaking elongation since the molecular orientation and crystallinity of the polymer are higher. Last, the filaments are gathered, lightly twisted, and stored on "beams" [13].

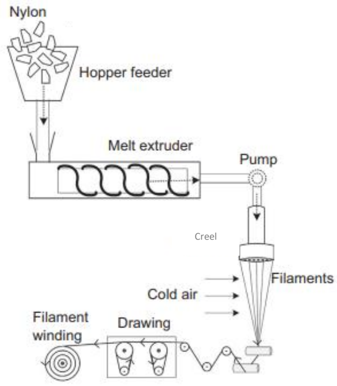


Figure 6. Nylon filaments production by melt spinning (Adapted [13]).

In contrast, the solution spinning (Figure 7) is done for polymers that don't have a defined melting temperature and must be dissolved for extrusion as continuous filaments. This concentrated solution is pumped through the creel immersed in a spin bath to obtain the fibers and remove the solvent. The filaments are heat-set to obtain highly oriented fibers and are not extensively drawn, since crystallinity and orientation are already highly developed. Then, as in the melting spinning, the filaments are lightly twisted, and stored on "beams" [13].

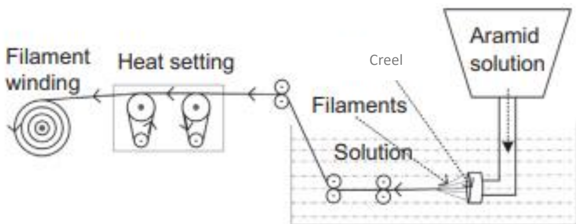


Figure 7. Aramid filaments production by solution spinning (Adapted from [13]).

After this process, the filaments are removed from the beams and twisted on themselves to a defined number of turns per inch (ply twisting), usually in the "Z" direction. Following that, two or more yarns are twisted in the "S" direction to form a greige (untreated) cord (Figure 8). Tire cords are usually balanced with equal twist levels in yarns and cords, and they can be constructed with different sizes and strengths [5, 6].

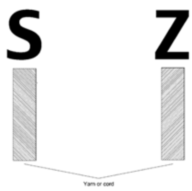


Figure 8. Twist direction (Adapted from [12]).

Figure 9 summarizes the cord construction process.

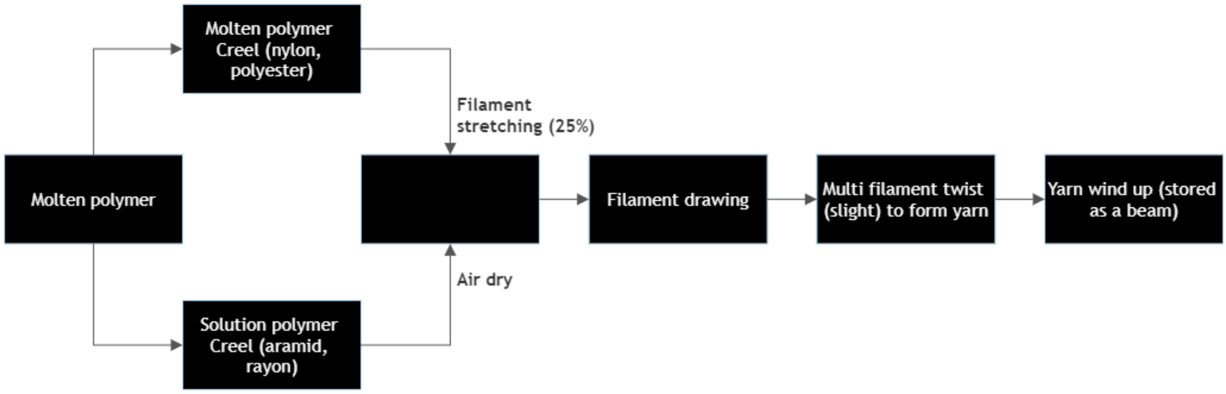


Figure 9. Cord production process (Adapted from [6]).

There are several advantages of using twisted yarns, most of them related to their strength, weight, flexibility, and elastic behavior. Firstly, twist levels in a tire cord are important with the regarding with its performance. In higher levels, the cord behaves like a spring, not opening up under compression, while in lower twists, the cord behaves like a rod, maximizing its strength. Besides that, the twist provides durability and fatigue resistance to the cord [6, 12, 17].

After the twisting, the cords can be woven into a fabric using small fill (pick) threads. The pick maintains the spacing between the cords uniform even after the downstream operations. A uniform cord distribution is essential for tire uniformity and performance. The number of cords per decimeter or EPDM is also important. High-end-count fabric provides higher plunger strength or penetration resistance, meanwhile, low-end-count fabrics have more rivets and have better rubber penetration around the cords [5, 6, 12].

2.2.3 Dipping process

Carrying on with the process, comes one of the most crucial parts of the process, the adhesive promotion coating (dip) treatment. This process is responsible for providing the fabric adhesion for bonding to rubber, optimizing strength, durability, growth, and elongation of the cord, and stabilize the fabric. The process consists of a series of steps to which the material is subjected. The adhesive process is a two-step procedure that starts with the adhesive application (dipping process) that is followed by a drying and by a heat treatment step. Then, the procedure is performed again previous to a final cooling step [5, 6].

For rayon and aramid cords, which are thermally stable and do not change significantly in downstream operations, this process is relatively simple. In addition, rayon must be carefully protected from moisture regain at all stages, to avoid uneven shrinkage across the fabric roll. The process starts with the rayon being dipped in a cord adhesive under relaxed conditions, to open the twist for a good dip penetration. After, the cord is tensioned to achieve the specification twist and dried at 130-150 ºC before being heated under tension at 155-175 ºC to cure the adhesive. In the case of aramid, heat treatment is at 230-260 ºC and generally at low tension and with very low stretch to standardize modulus [6].

Oppositely, for nylon and polyester cords, which are thermoplastic fibers, if the cord processing is carried out below crystal melting temperature, it modifies the non-crystalline portion. In the usual process of dipping and baking a cord adhesive, the amorphous portion of the cord tends to become less oriented, resulting in a shrinkage that will adversely affect tire uniformity. Stretching the cord in the first heating zone and then allowing the cord to relax in a controlled way in the second heat treatment zone will control shrinkage. The heat treatment for this type of material then is done between around 170 ºC and 250 ºC. Nylon is generally used at 3-7% net stretch, and polyester at 0-4%.

This process aims to avoid separation at the cord-adhesive interface, at the rubber-adhesive interface, or within the adhesive itself.

2.2.4 Rubberizing manufacturing process

The following step is rubberizing the cord or fabric. It can be done by a calendering process for the fabric structure, where is produced a cord-rubber sheet or by a cap strip line for the SEC, in which cap-plies are produced.

The cap-ply is a strip of cord impregnated with rubber compound (matrix) produced by a cap strip line machine, that is wrapped around the tire, forming the textile ply. The cap strip line starts with storage where the textile cords spools are positioned and tensioned. After, the cords, fed from a creel, pass through an extruder and a head, which is connected to rubber compound flow channels that allow the rubber to be incorporated into the cords forming the strips. After, the strips need to be cooled down rapidly to allow a correct winding step. The temperature control before the winding step is extremely important to avoid the adhesion between the wound strips that would lead to exposure of the cords (separation from the rubber) during the unwinding process [8, 18, 19].

As for the rubber sheet, the calender machines are used. Calenders press the rubber compound between two or more rotating rolls forming thin flat sheets with a precisely controlled thickness that are coated onto fabric on both sides [1, 3, 5, 6].

In component preparations, there are three types of calenders: gum calenders, which make rubber sheets, such as inner liners; fabric-coating calenders, where the rubber is incorporated into the fabric and steel cord calenders [5].

These machines are equipped with three or more chrome-plated steel rolls that revolve in opposite directions. The rolls are heated with steam or circulating water and can operate at variable speeds and temperatures to obtain the best processing conditions [1, 5].

In the case of fabric calendaring, the fabric passes through the calender rolls, and the compound is applied on both sides to a complete cord's topping. It is important to emphasize that rayon and occasionally nylon fabrics need to be dried prior to entering the calender. Usually, this is achieved by passing the fabric over a set of heated rollers, at temperatures of around 80-130 ºC, depending on the material [12]. After that, the sheet is cooled down in a water tank and usually is powdered with an anti-tack agent before a cooling and drying festoon. In the end, the material is placed in a space with a target temperature below 40 ºC [5]. A rubberized cord sheet of the specified thickness and width is formed at the calender output.

In the calendering process, several parameters are under control to obtain the optimum results. First, the compound to be applied must have a satisfactory temperature and plasticity, which are controlled and reproducible from batch to batch of material used. Also, the feed of the compound must be precisely controlled: a feed too high would lead to an increasing rolling bank at the calender meanwhile, a feed too low would give poor calendaring with uncovered areas and uneven thickness. Secondly, the temperature of the rolls must be controlled to give uniform coverage and surface smoothness. Furthermore, the thickness of the rubber applied must also be controlled to be the same for the whole extension of the sheet [12, 20].

At last, the approval of the manufactured is performed by quality control testing against product specifications.

2.3 Reinforcement tests

Since tire cords have a huge contribution to the performance of the tire, to achieve all the required conditions, it is necessary to perform a specific characterization of the material. The mechanical and physical testing ensures that the material acts in accordance with the performance requirements of the industrial application. The materials' tests are done in the Reinforcement Testing Laboratory under a predetermined certification in which the arrival, the conditioning, and the test situation are given. This guideline is important because it can constrain most of the factors that might affect the test results as the sampling, the atmospheric condition during testing, methods of testing, and instruments used [21].

For the evaluation of the mechanical properties of the materials two types of tests were studied in the present work: tensile test and twist level test.

Tensile testing is a destructive test procedure that measures the force required to rupture (break) the material and the extension of which the material is stretched or elongated to that breaking point. It provides information about the tensile strength, tensile modulus, tensile strain, and elongation in percent. This is a relatively simple test whereby the material is pulled until it breaks. The results can be given by a stress-strain curve. Polymers can exhibit a vast range of mechanical behavior depending upon the conditions the test is performed. A typical stress-strain curve for reinforcement fibers is demonstrated in Figure 10 [22, 23].

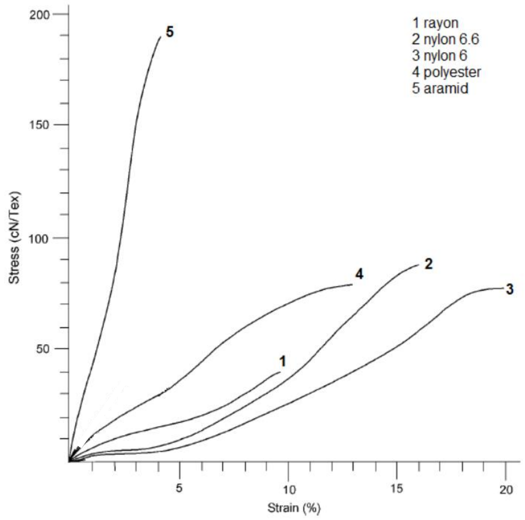


Figure 10. Stress-strain behavior of textile fibers (Adapted from [12]).

The twist level test is also a destructive process in which the cord is untwisted to count the number of twists per unit length. A cord with a known length is untwisted using a Twist Tester by rotating one end of the specimen concerning the other until the yarns of the tested cord are parallel [24]. The optimum twist level depends on the intended utilization of the cord since twist affects both the strength and elongation properties (Figure 11).

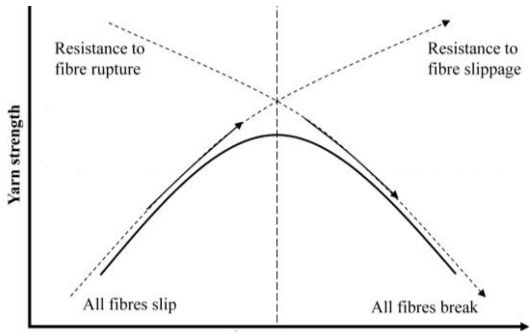


Figure 11. Twist level and breaking strength relationship (Adapted from [25]).

However, even though, with the increase of the twist, the durability and the breaking strength also increase, there is an optimal twist level, where from it the breaking strength decreases independently of the type of material (Figure 12). The increase in the strength of the cord with the twist results from the increase in the cohesion of fibers as the twist is increased. As the twist keep increasing, also increases the helix angle of the cord or the angle between the filament axis and the cord axis, reducing the effective contribution to the axial loading of the yarn due to fiber obliquity [17, 26, 27].

In this way, the strength-twist relationship is important because it determines the optimum twist to the optimum strength, thus tire cord twist level must be kept in a specified range.

3 Materials and Methods

This chapter will present all the materials and methods used in the development of the study to accomplish the proposed objectives. This section will demonstrate the equipment, test procedure, and parameters used/analyzed to accomplish an optimal procedure that can guarantee reliable and repeatable results.

The first part contains the study regarding the sample preparation and conditioning methods to improve them. The test procedures were done in accordance with test instructions established by the company where the study was performed, which follow ASTM and ISO standards. However, in some tests several parameters were analyzed in opposition to current guidelines to study their influence on test results.

The second part of the study is an additional analysis done by a coordinated cross-check testing between different laboratories. The concept of a cross-check testing is to verify the comparability of a test performed in multiple laboratories. Hereby, the tests are done with the same materials at the same conditions.

3.1 Materials

The materials supplied for the tests as well as their specification is presented in Table 3:

Table 3. Materials' specifications.

Material	Cord structure	Construction	Status	Twist level / tpm	Material code in this study
Hybrid (Aramid + Nylon 6.6)	SEC	1680dtex + 1400dtex	Dipped	370	H1
			Rubberized		
Nylon 6.6	SEC	470dtex x2	Dipped	420	N1
			Rubberized		
		1400dtex x2	Dipped	290	N2
			Rubberized		
Polyester	Fabric	1100dtex x2	Dipped	410	P1
			Rubberized		
		2200dtex x2	Dipped	300	P2
			Rubberized		

The dipped SEC materials are supplied in a spool. The rubberized SEC comes in the cap strip form, where it is wounded up with a varied thickness depending on the material, but in 1 cm wide. The textile fabric comes with a width of approximately 2 m with a length of 1 m. The rubberized fabric materials (calender sheet) are also supplied with a varied thickness, but with the entire calendar width with a length of approximately 1 m.

3.2 Test procedure and conditions

3.2.1 Samples' preparation and conditioning

The samples' standard conditioning is guided by ASTM D1776 in which all the samples must be conditioned for at least 24 h prior to testing in the standard atmosphere defined as [28]:

Temperature	(23 ± 2) °C
Humidity	(55 ± 5)%

Dipped fabric materials were conditioned on a flat surface and then cut at the testing time. Dipped SEC materials were conditioned on the spool and then tested directly from there.

When testing SEC material, a length of 25 m was discarded before testing in order to ensure that the material is tested with same condition (pre-tension).

Whereas the rubberized material, the cords require to be prepared out of the rubber matrix before being tested. As concerns the rubber sheet, 5 cm width from the edge of the sheet must be removed and discarded. After, another strip is taken in order to facilitate the cords pull out. For both sheet and cap strip, the strip is torn between two textile cords, and the thread with the lower cover of rubber is used. The cord then is conditioned for 24 h on a flat surface with a knot in the end to avoid twist loss.

The several preparation and conditioning parameters investigated are described in Table 4.

The *manner of preparation* is with regards to how the samples were handled, namely regarding the status of the hand during the preparation. The *position of the cord* is specifying where in the rubber matrix and how the cords are pulled out. The *speed* is concerning the velocity in which the cord is pulled out of the compound. At last, the *method of conditioning* is how the samples are relaxed before testing. In order the support the understanding, Appendix B - provides figures of the methods.

Table 4. Description of preparation and conditioning parameters.

Phase of testing	Parameters	Name	Description	CODE
Samples' preparation	Manner of preparation*	Bared clean and dry hands	The hands were washed with soap and dried with paper or towel.	CLEAN
		Hands with cream	Between 5 and 10 minutes before the samples' preparation, hand cream was applied in both hands.	CREAM

Phase of testing	Parameters	Name	Description	CODE
		Sweaty hands	Simulation of sweaty hands by spraying water in the hands before the handling and testing.	SWEATY
		Rubber gloves	Gloves were used during the preparation and the testing.	RubG
		Cotton gloves		CotG
	Position of pulling the cord	Random preparation	The cord is taken by the easiest way to pull out of the rubber matrix without any rules.	RP
		Easy preparation	A strip is taken, torn in the middle, where a cord gets free, the two new strips are pulled apart from each other until the end.	EP
		Difficult preparation	Calender: The cord is taken from the edge of the strip. Cap strip: Only rubber is pulled out from the edge, and the cord closer to the edge goes out freely.	DP
		Pulling only cord preparation	Pulling only the cord without tearing apart the rubber strip. A small tear is made only to have an end of the cord out to pull.	PCP
		Fixed end preparation	One of the ends of the strip is fixed and the strip is torn apart to get more cords as possible.	FEP
	Speed of preparation	Slow (500 mm·min^{-1})	Used the tensile machine with peel test clamps to pull out the cords.	SLOW
		Fast (1000 mm·min^{-1})		FAST
Samples' conditioning	Method of Conditioning*	Hanging	The samples were hung.	H
		Hanging with a knot	The cords were hung with a knot unifying both ends.	H/N
		Laying	The cords were put in a flat surface.	L
		Laying with a knot	The cords were put in a flat surface to condition with a knot unifying both ends.	L/N

*The RP is used as a preparation reference of how is normally done in the laboratory, therefore it is the procedure used in 'manner of preparation' and 'method of conditioning' parameters.

3.2.2 Cross-check testing

The second part of the study is a coordinated cross-check testing between different laboratories. Both dipped and rubberized material were analyzed. Samples came from identical plant and production lot (before and after passing through the rubberizing process).

Force-elongation and twist level were tested. The dipped samples were tested as the current guidelines state, but also for H1, the samples were tested conditioned on a flat surface. The rubberized samples were tested directly after pull-out and conditioned according to the current guidelines. They were delivered in the same conditions to the different laboratories as well as the data report forms and test instructions. Each operator performed the tested in the normally conduct – specified by test instruction.

3.2.3 Tensile tester

A ZwickRoell tensile tester Z005 was used for the tensile measurements (Figure 12).

Figure 12. Tensile test machine.

The tests were carried out in accordance with test methodologies established by the company which follows the ASTM D885 standard.

Each sample consist in a piece of cord – at least 200 mm longer than the clamping length – that is clamped in the tensile testing machine and then stretched until broken. The stretch is done by moving the lower grip with a constant speed until the material's rupture. The breaking force, elongation and EASL or LASE (see in Appendix A -) are determined directly. The materials' specification is done only in particular parts of the curve. The data taken from these parts comes with mean values and standard deviation (SD). The specification for each material used is described in Table 5.

Table 5. Materials' specification for force-elongation tests.

Material	Specific measurement	Values	Pre-tensioning / cN
H1	LASE	1% 3% 4% 7%	154
N1	LASE	2%	47

Material	Specific measurement	Values	Pre-tensioning / cN
N2		4% 8% 12%	210
P1	EASL	45 N 90 N	110
P2		45 N 90 N 180 N	220

The results analysis is done comparing these defined values per material, with regards to mean values, standard deviation, and the coefficient of variation (CV).

The number of samples tested in each test was 5, except for the cross-check testing, where 10 samples were tested for each.

3.2.4 Twist Checker

A Zweigle Twisted Checker type D321 is used for the twist count (Figure 13).

Figure 13. Twist level test machine.

The tests were carried out in accordance with test methodologies established by the company which follows DIN EN ISO 2061, in which, besides the amount of twist, the determination of the direction of twist in yarns and the change in length on untwisting is also provided. The cord is fixed between two clamps: one fixed and one rotational. For a specific length (500 mm) and cord pre-tension (the same as for the force-elongation tests), the rotational clamp is turned against the twist direction of the cord until the yarns are parallel. The number of turns is recorded on the counter. In this study only twist in S-direction was observed. Just as the force-elongation tests, the number of samples tested in each test was 5, except for the cross-check testing, where 10 samples were tested for each. Also, the criterion of analyzing the results concerns the mean values, their SD and CV.

3.3 Test results calculation and analysis

With respect to the force-elongation curves, the data for each curve (each specimen) is collected and interpreted by the applied test software in which the breaking force, elongation at break and EASL or LASE are determined directly and a mean curve is designed. The SD within the specimen curves in the sample is calculated to measure how widely the values are dispersed from the average value (the mean). The target is that the SD is as low as possible, giving the test repeatability. Besides that, when comparing two or more mean values, if the SD of two mean values overlap, one can say that these mean values are not distinguishable [29].

However, the objective is to find the better preparation method, therefore it was also compared the FASE or EASL mean values and the twist level mean values within the preparation methods to find which preparation method was significantly deviating from the ‘normal’ behavior and which one is the best approach to follow.

Thus, the results were analyzed and ranked with the following priority of selection:

1. Mean value of different preparations: the comparison of how the mean values behave together is done in terms of the standard deviation values.
2. Coefficient of variation of each preparation (CV): the ratio of the standard deviation to the mean. It is given in percentage.
3. Easiness of preparation: a personal evaluation with respect to how the preparation was done.

For instance, if the mean curves don’t show any significant difference within each other, that is if the SD values of different preparation are overlapping, the second priority is verified. If the same happens again, the easiness of preparation advises on the choice of the best preparation method for each material.

4 Results and discussion

4.1 Samples' preparation and conditioning

In this part of the chapter conditioned sample results are shown. The analysis is done comparing the results of the cords after they are pulled out of the rubber matrix. The dipped (reference) curve represents the material behavior before preparation and rubberizing processes. In this first part, N2 was not tested. Graphs that support the analysis of the force-elongation results are presented in the Appendix C -.

4.1.1 Manner of preparation

The first consideration was regarding the manner that samples were handled. In this case, five different ways of handling the samples were executed to study their influence in the test results and practice. The results presented are referred to conditioned samples.

The first material to be analyzed is H1. The force-elongation behavior, twist level results with their respective SD, and their CV values are presented in Figure 14. The CV limits represent the specification limits in which the results should be under to be considered reliable.

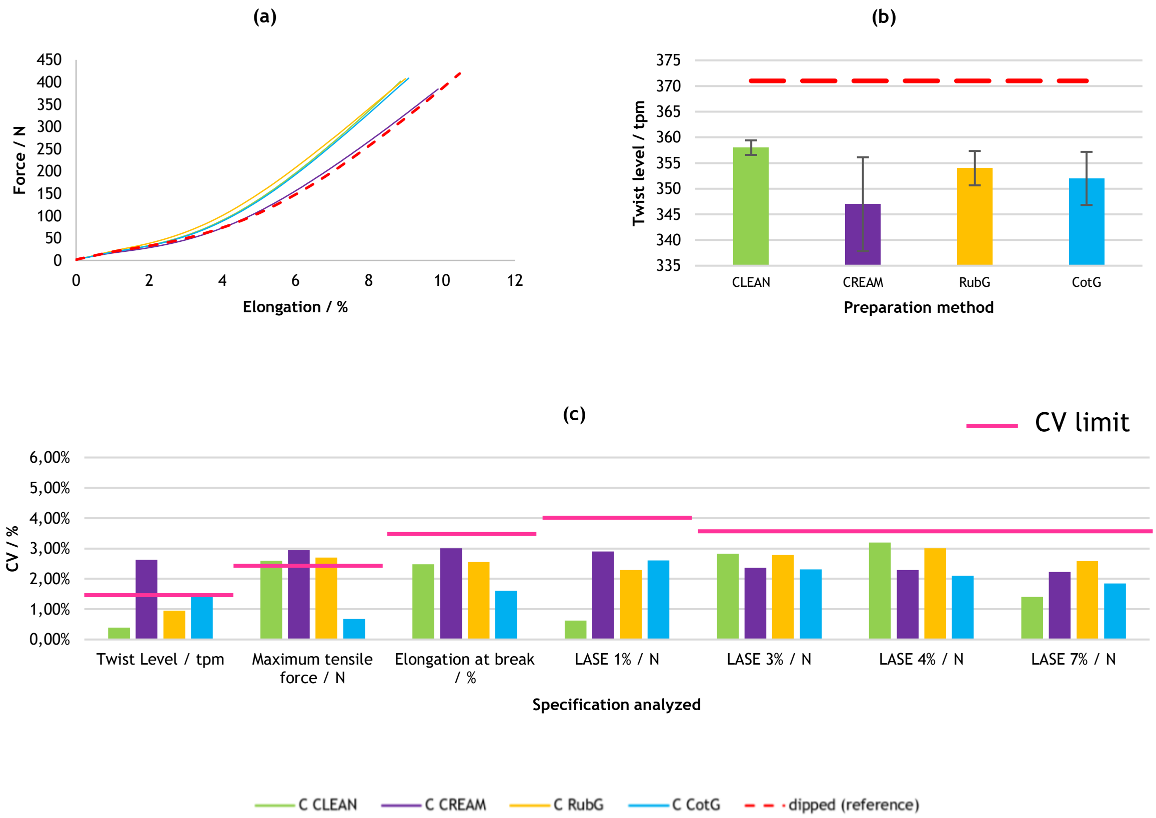


Figure 14. H1: (a) force-elongation curves, (b) twist level test and (c) CV values.

From the force-elongation curves (Figure 14.a), it can be observed two groups of curves that are comparable: RP, RubG and CotG, as well as CREAM and dipped (reference). After rubberizing and preparing, one has three curves highly comparable, but distinct from the reference curve, while one curve (CREAM) is closer to the reference but different from the other three preparations. The hypothesis in this case is that that the rubberizing process has an impact on the material, resulting material properties changes (offset) compared to the reference curve. With the graphs available in Appendix C -, it is possible to visualize that the CREAM preparation significantly differs from all the curves in the LASE 1% and LASE 3% specifications, as well as in the lower maximum tensile force.

Moreover, CREAM is the one losing more twist during the preparation (Figure 14.b), presenting a significant difference when compared to CLEAN preparation, the one with the most preserved twist level. RubG and CotG also show lower twist level values, but they are still comparable. This sensitivity of H1 is evident in the contrast between the dipped and rubberized materials, as well as among the different preparations. This can be explained by the distinct properties of the different materials used, as the yarn diameter and bending stiffness which contribute to the formation of a less stable cord. Besides that, aramid's crystalline configuration could also be a contributing factor. Aramid fibers are highly oriented with a very high degree of crystallinity, making them more susceptible to being unsettled by twist [30].

Regarding the CV of both tests, there is not a tendency of low values for a specific preparation. CREAM presents high CV on twist level, exceeding the established limits. Besides that, it also exhibits CV above limits in maximum tensile force, along with CLEAN and RubG.

CotG presented a stable and comparable force-elongation results despite being one of the most difficult preparations after CREAM. In both preparations occurs the loss of the fingertip grip to tear rubber without slipping and difficulty to do a knot in the ends for conditioning.

In a summary, the preparations can be classified by green, representing the preparation recommended; yellow, preparation that didn't show a significant positive or negative impact onto the results, and red, preparation to be avoided. Applying this classification to H1, one has the following:

The SWEATY analysis for H1 is explained and described in Appendix D -.

The next material to be analyzed is N1. Figure 15 presents the test results as well as their CV. As for H1, it can be an offset between dipped and rubberized in both force-elongation curve (Figure 15.a) and twist test results (Figure 15.b) for N1.

In the force-elongation, the curve behaviors are distinguishable. RubG and CotG have showed similar and comparable values among themselves but different from the other preparations.

CLEAN and SWEATY yielded similar results overall, except for the maximum tensile force specification. In this aspect, CLEAN exhibited a lower value, making it comparable only to CREAM.

In terms of twist level (Figure 15.b), RubG and CotG showed significantly lower values compared to the other preparations, approximately 10 tpm less than the most conserved twist level preparation (CLEAN). This difference may be attributed to the challenge of knotting the ends for conditioning with these preparations. Additionally, the gloves used were not the ideal size, which hindered knotting due to the fingertip getting in the way. Furthermore, since a very thin cord (the smallest construction) was used, it tended to slip multiple times.

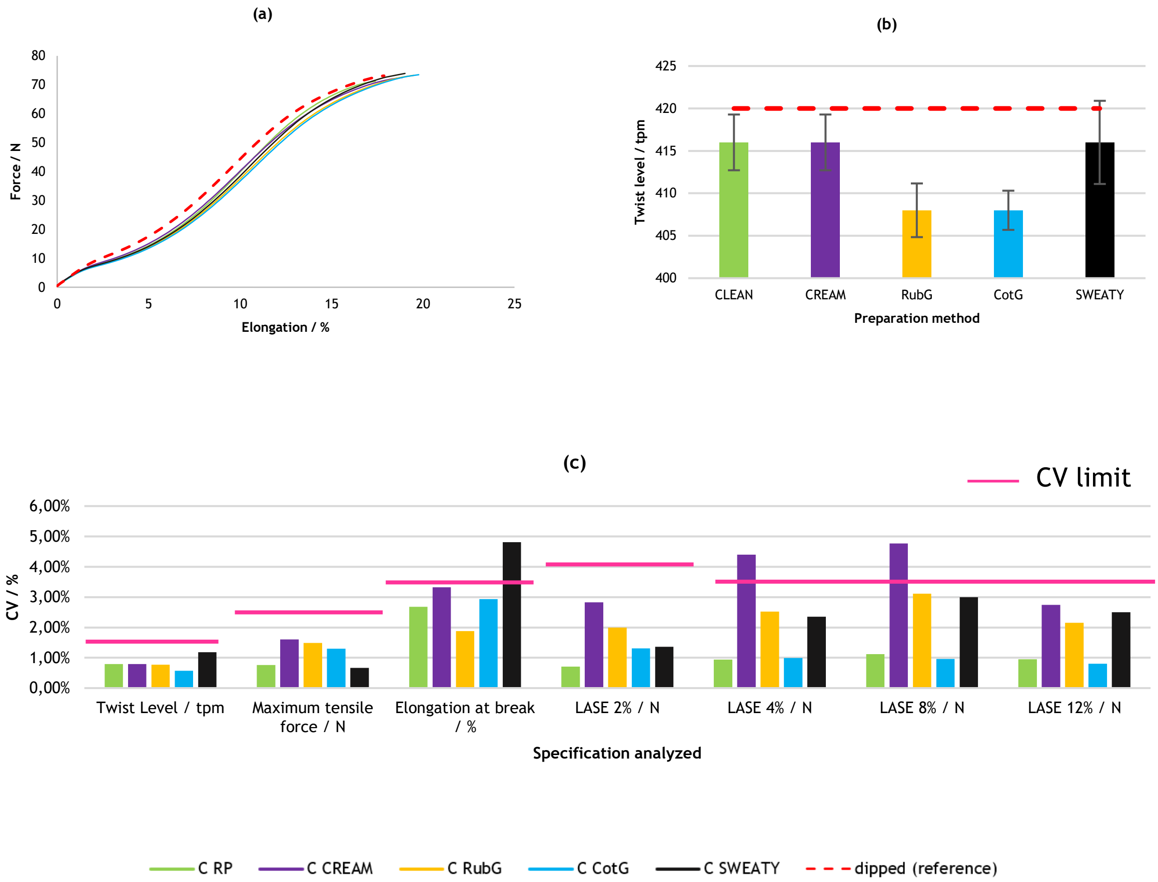


Figure 15. Test results for N1: (a) force-elongation test, (b) twist level test and (c) CV of manner of preparation test results.

As for the CV of both tests, CLEAN and CotG preparations exhibited an outstanding repeatability having values below 1% for most of the specification analyzed. The only exceptions were elongation at break, LASE 2% for CotG, and LASE 8% for CLEAN, but even these values are under the established limits (Figure 15.c). In contrast, CREAM and SWEATY exhibited the opposite behavior, displaying some of the highest CVs across almost all specifications. As for RubG preparation, while it demonstrated a stable performance in terms of repeatability in both tests, it did not exhibit any remarkable features. However, it did show a higher tendency to lose twist level more easily compared to other preparations. The CLEAN preparation would be the safest choice for this material namely because of its CVs.

For the first polyester cord, P1, the different handlings did not have a significant impact on the force elongation test results (Figure 16), except in elongation at break, where a comparison between CLEAN and, CotG and RubG was not possible. Furthermore, it is not observed the same offset between dipped and rubberized materials, as was seen previously for H1 and N1.

Note that twist loss was observed in the CotG preparation, as evident from Figure 16.b. Although the twist loss is approximately 5 tpm, resulting in a loss of less than 1%, it represents a significant difference since the SD range do not overlap. Despite experiencing twist loss during preparation, the CotG handle demonstrates one of the lowest coefficient of variation values for the twist level test, approximately 0.30%.

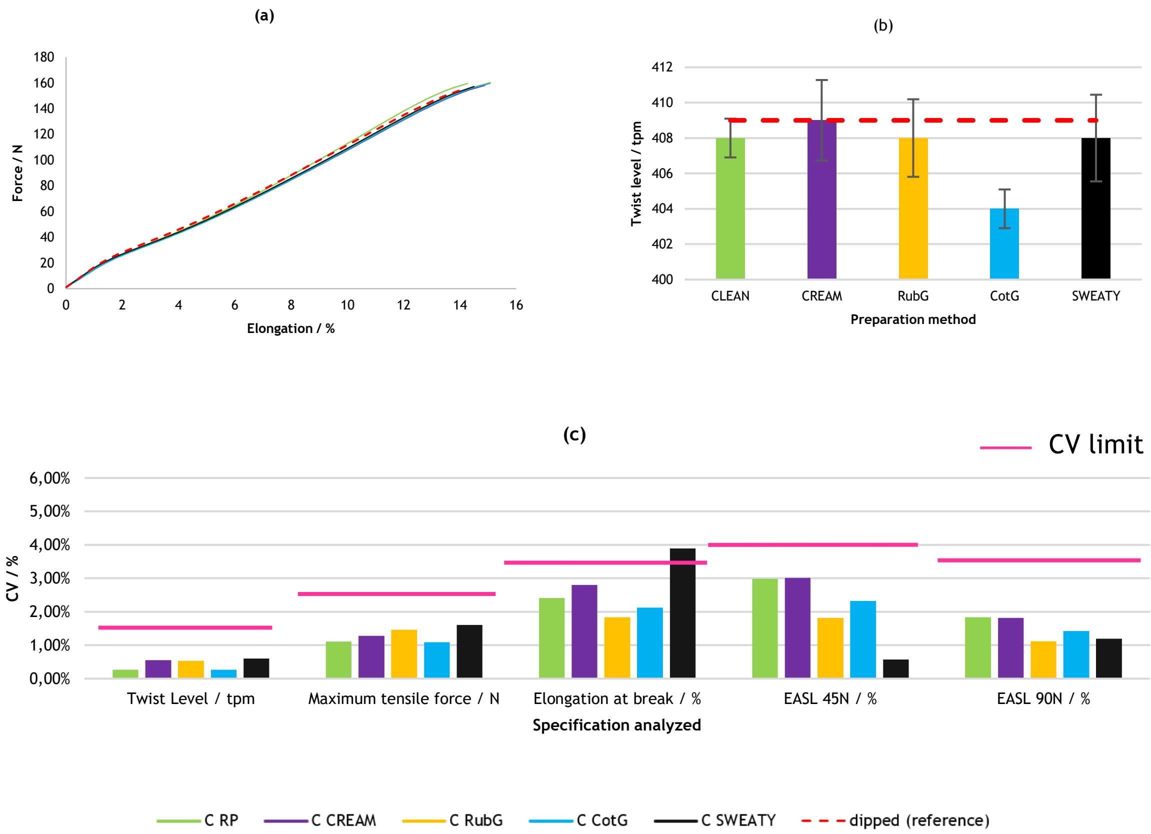


Figure 16.Test results for P1: (a) force-elongation test, (b) twist level test and (c) CV of manner of preparation test results.

It is possible to affirm that the manner of preparation does not significantly influence the outcomes. The results obtained demonstrate highly acceptable and comparable results, with the exception of CotG in the twist level test. Therefore, the recommendation for this material would be to avoid using CotG in its preparation.

Finally, in the case of P2, it was observed that the handling had a significant impact on the force-elongation test results (Figure 17). Additionally, a slight offset between the dipped and rubberized materials was observed. Notably, the preparations that involved no physical contact with the material (CotG and RubG) exhibited similar behavior, while the preparations with higher moisture content (CREAM and SWEATY) displayed a similar trend. The CLEAN preparation fell in between these two trends.

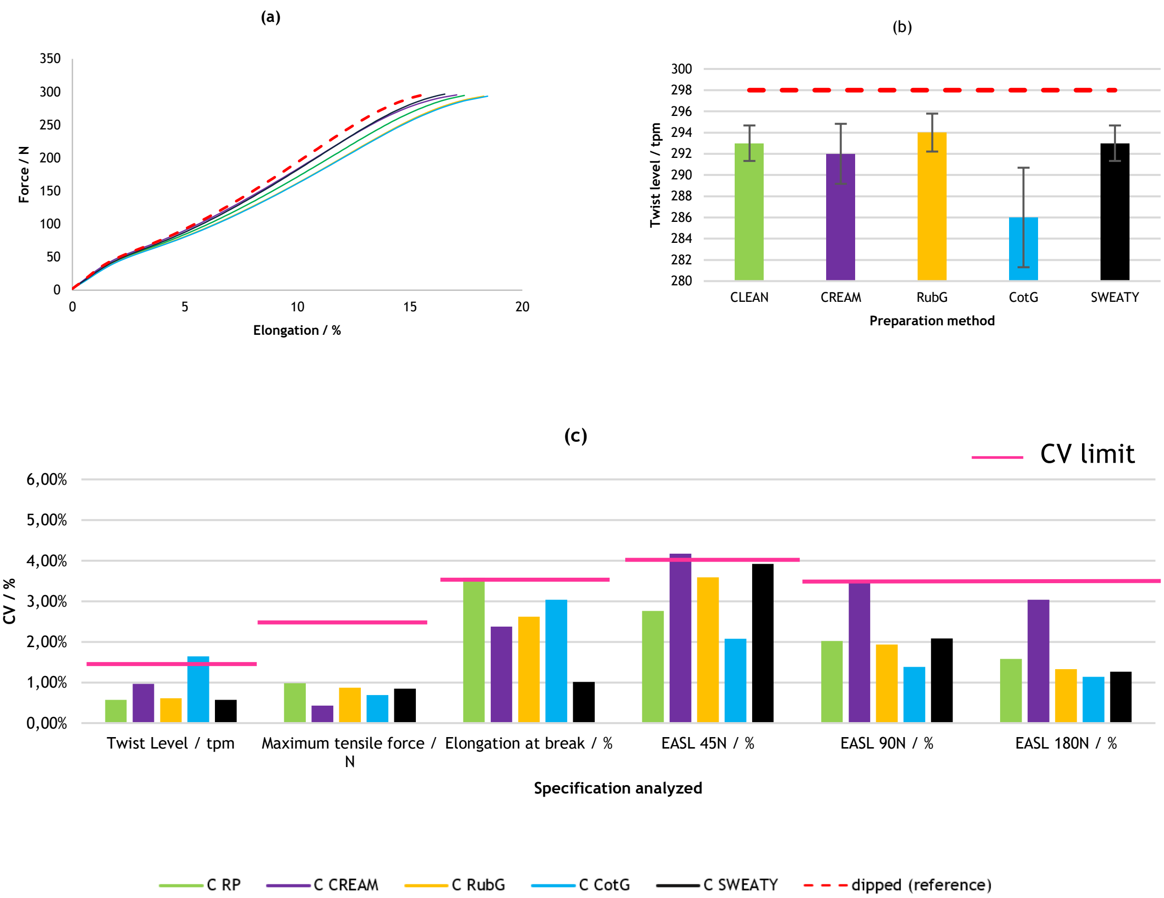


Figure 17. Test results for P2: (a) force-elongation test, (b) twist level test and (c) CV of manner of preparation test results.

As expected, CotG was the one preparation losing more twist during the handling (Figure 17.b), about 8 tpm resulting in 2.7% of loss. Furthermore, CotG was the most impacted in terms of the CV value among all preparations, surpassing the established limit for twist level (Figure 17.c).

Regarding the CV values, in addition to the aforementioned observations, CREAM preparation also exhibits notable proximity to, and occasionally surpasses, the limit in certain specifications.

Furthermore, it was not identified any trend of best preparation when both tests are analyzed together. When one analyzes the specifications separately, it is possible to observe that for twist level, CLEAN, RubG and SWEATY demonstrated similar and lowest CV values. On the other hand, for force-elongation, the preparation with the lowest CVs was CotG.

Hence, RubG would be one of the best approaches with this material. It exhibits a moderate behavior when compared to the others' reproducibility, without exceeding any CV limits, and it does not provoke significantly twist lost during its preparation.

4.1.2 Position of preparation

Cord positioning in the rubber matrix and preparation procedure have been compared with regards to the analysis results. The results presented are referred to conditioned samples. Figure 18 illustrates H1 behavior in terms of force-elongation (Figure 18.a), twist level (Figure 18.b) and its CV values (Figure 18.c).

Again, a notable observation is the offset observed between the dipped and rubberized materials in both the force-elongation and twist level tests. Additionally, it is noted that when analyzing the studied preparations, it is apparent that they form a single group of curves. However, within this group, two preparations, PCP and EP, deviate towards the edges. However, the deviation of EP is not significant, whereas the deviation of PCP is more notable.

In terms of the twist level (Figure 18.b), the two mentioned curves, EP and PCP, exhibited the highest degree of twist loss and FEP the most preserved twist level. Particularly, PCP experienced a twist loss of over 20 tpm, equivalent to approximately 6%, which in this case is relevant. During this procedure, extracting only the cord from the rubber requires greater effort. Additionally, it appears that the compound of the cap strip, being more elastic and resistant to disintegration compared to the calendered material, causes the rubber to slip through the cord, resulting in difficulties during the procedure. This clearly impacts the cords and contributes to the observed twist loss.

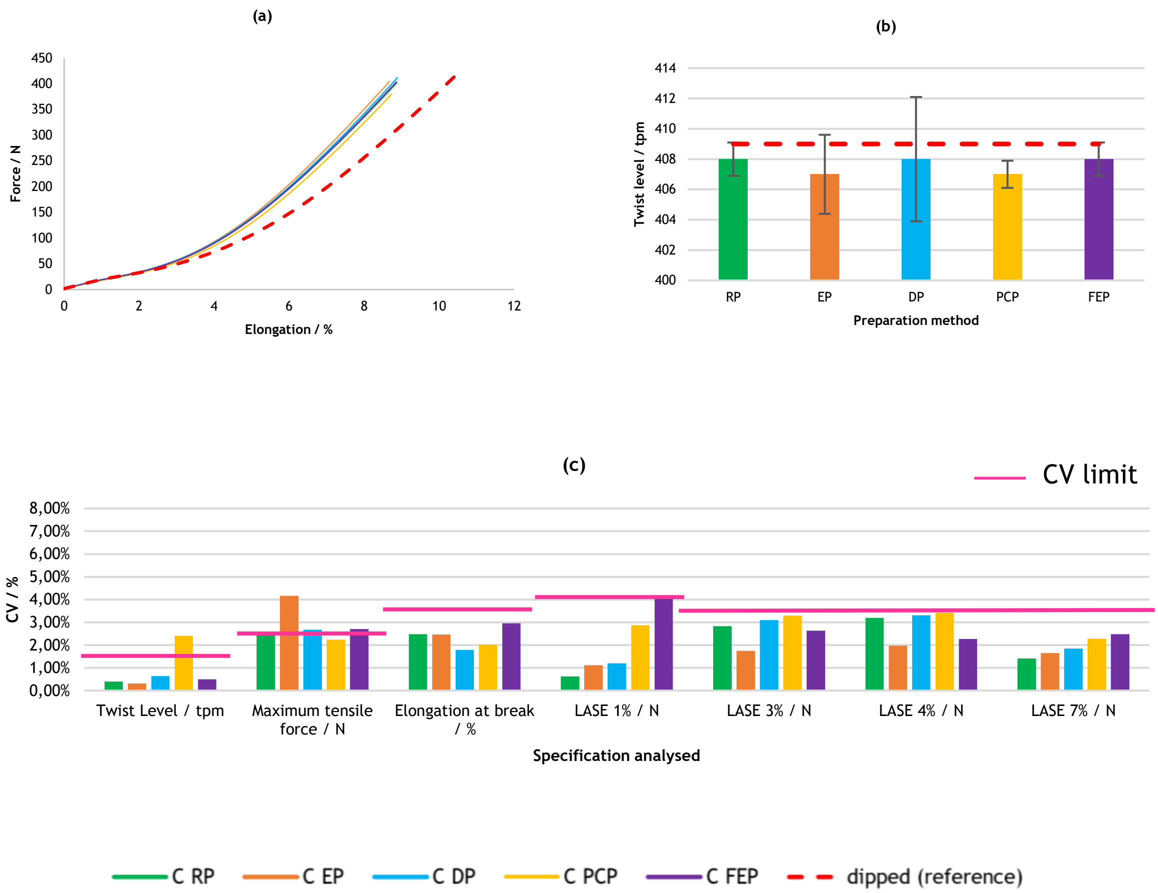


Figure 18. Test results for H1: (a) force-elongation test, (b) twist level test and (c) CV of position of preparation test results.

As for CV values, it is evident that there are higher CV values compared to the previous parameter analyzed for the same material. Specifically, PCP consistently exhibited values near or above the established limits. On the other hand, despite having a CV value almost double the allowable limit in maximum tensile force, EP demonstrated one of the lowest CV values for the other force-elongation specifications.

Based on the analyzed results, it is recommended to avoid using the PCP procedure with this material. By contrast, the RP and DP preparations should be strongly considered as they exhibit CV values below the established limits.

Carrying on with the analysis, N1 test results and respective CV values is presented in Figure 19.

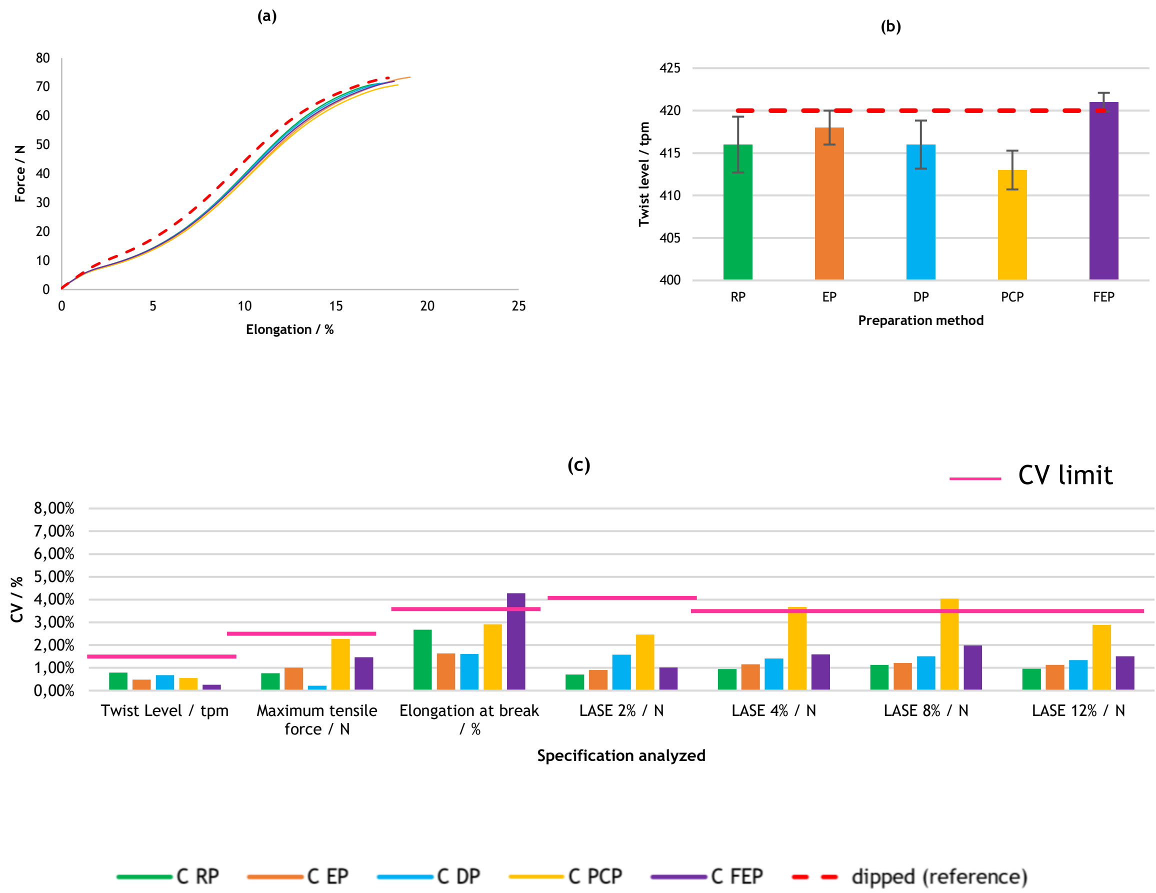


Figure 19. Test results for N1: (a) force-elongation test, (b) twist level test and (c) CV of position of preparation test results.

In the force-elongation analysis, the curve behaviors initially start off similarly with comparable values (Figure 19.a). However, as the curve progresses, an offset from the reference curve becomes evident. Additionally, a slight deviation is observed in the curves of PCP and EP compared to the other preparations, similar to what was observed for PCP in H1.

Both materials H1 and N1, which are presented in cap strip configuration, were notably affected by the PCP preparation. The configuration of the material made the procedure more challenging, ultimately impacting the test results as previously explained for H1.

Regarding twist, the PCP exhibits a significantly lower twist level compared to FEP, which is the preparation that best preserves the twist. However, the twist level of PCP remains comparable to o RP and DP.

In terms of CV values, PCP shows poor repeatability, as it has significantly higher CV values than the established limits.

Despite the aforementioned observations, in most force-elongation specifications, there is a consistent trend of lower CV values for RP and EP. FEP, however, is having in most specifications the second highest CV value, but the most preserved twist level – with the lowest CV value for this test.

According to the results analyzed, the only procedure recommended to be avoided with this material is PCP. FEP is a helpful procedure because avoid rubber slip through the cord, but it should be done always with the attention to elongation at break, if it is something that has a tendency to recur, it should be avoided and studied the root cause, because the other preparations present more acceptable results.

As regards to P1, the different positions did not significantly affect the test results (Figure 20). Except for EP, in which the initial modulus of the curve is lightly smaller and the maximum tensile force higher (the curve is slightly tilted to the left) (Figure 20.a).

Also, note that EP demonstrates the highest CV values in both EASL 45N and EASL 90N specifications, with the values exceeding the limit in the first one.

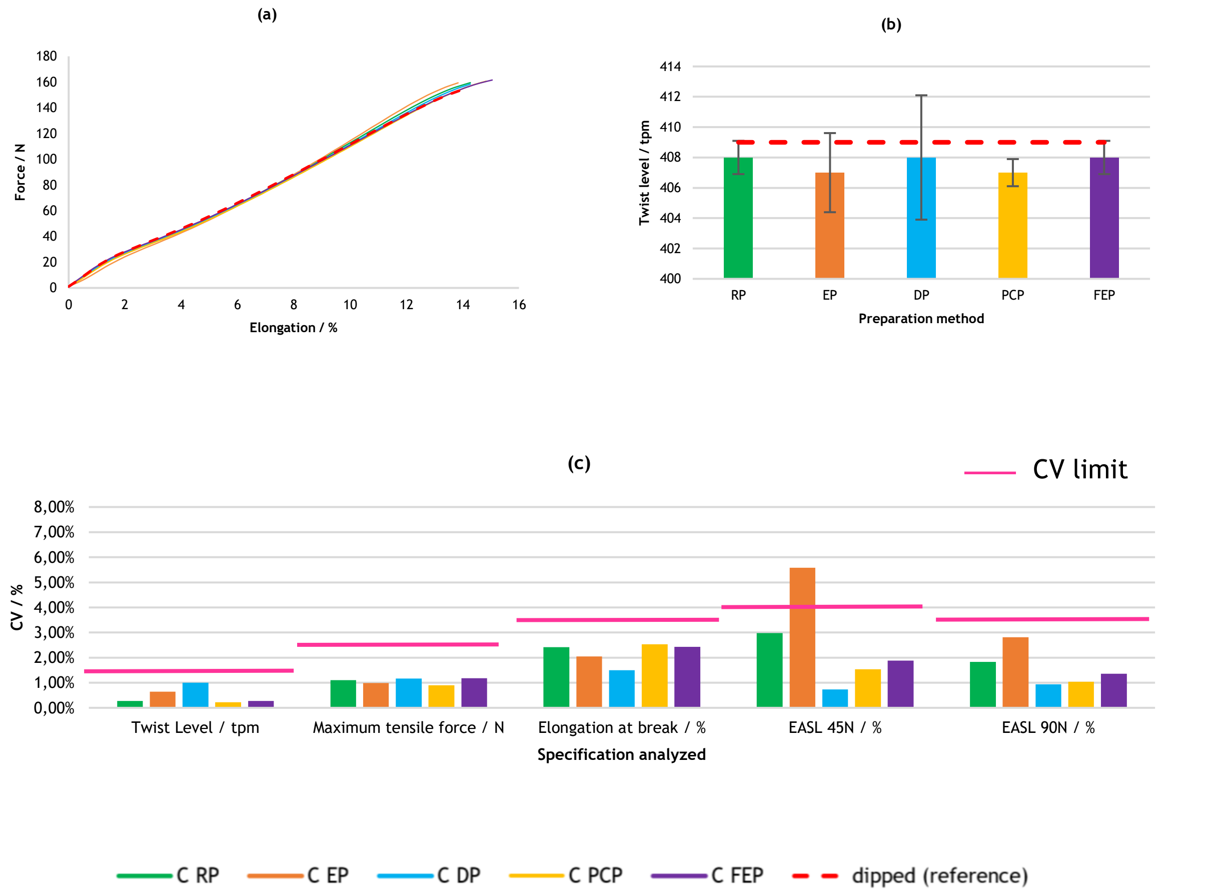


Figure 20. Test results for P1: (a) force-elongation test, (b) twist level test and (c) CV of position of preparation test results.

Disregarding the controversies above, no preparations should be avoided as all of them showed stable and comparable test results. However, DP, PCP, and FEP can be considered the most recommended options, as they exhibit a tendency for higher repeatability with lower CV values.

Finally, as occurred in subsection 4.1.1, P2 is highly influenced by the position of preparation in terms of force-elongation curves, as depicted in Figure 21. Similar to P1, the EP curve exhibits a smaller initial modulus and a higher maximum tensile force compared to other preparations, with the curve slightly tilted to the left. Additionally, when it comes to twist levels (Figure 21.b), EP stands out as the only preparation with a significantly lower twist level compared to the others, which have shown relatively comparable twist levels.

As for CV values (Figure 21.c), RP exhibits a CV value slightly above the limit for elongation at break, followed by PCP, which falls just below the limit. However, both PCP and FEP demonstrate high CV values in EASL 45N, surpassing the limit by approximately 50% and 100% respectively.

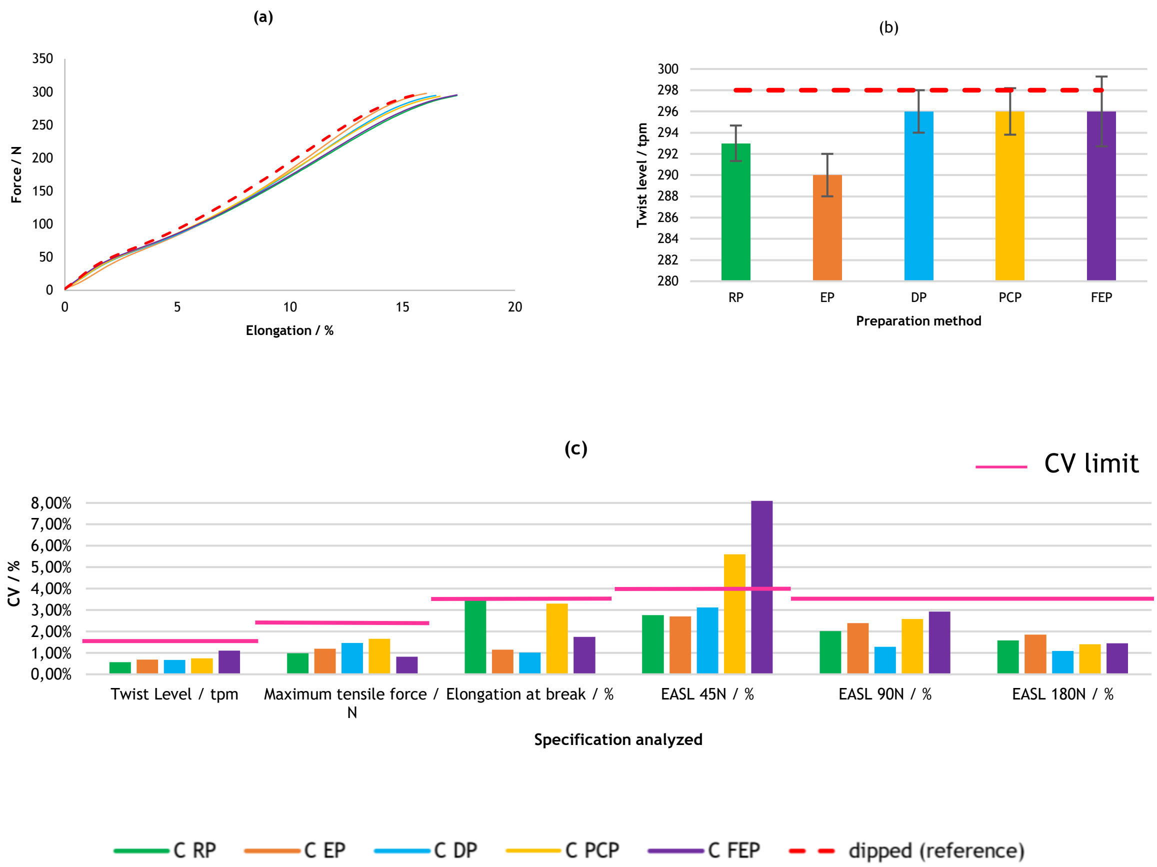


Figure 21. Test results for P2: (a) force-elongation test, (b) twist level test and (c) CV of position of preparation test results.

There is no clear trend indicating a superior preparation when both tests are analyzed together. However, EP should be avoided due to its distinct behavior in the force-elongation curve and significantly lower twist level. The preferred approach with this material would be to select a preparation that exhibits moderate and stable behavior, ensuring good reproducibility compared to the other preparations. In this regard, DP can be considered one of the most reliable options.

4.1.3 Speed of preparation

The effect of the velocity at which the cord is pulled out of the rubber matrix was studied. This procedure was conducted using the same tensile tester machine employed in the force-elongation tests, albeit with different clamps. The cords were extracted at a rate of 500 $mm \cdot min^{-1}$ for the SLOW preparation and 1000 $mm \cdot min^{-1}$ for the FAST preparation. The results presented are referred to conditioned samples.

As it is shown in Appendix A, cap strip and calendered materials were prepared in a different way. The calendered strip was placed on the top clamp and one of the ends of the cord was partially pulled-out and placed on the bottom clamp – simulating a PCP procedure. As for the cap strip, the strip was partially tear apart in the middle, and each new part was placed in one of the clamps – simulating EP. Starting with H1 test results and CV values, as is shown in Figure 22.

The different speeds of preparation tested had a minor impact on the force-elongation curve (Figure 22.a), particularly for FAST in the LASE 1% and LASE 7% specifications. However, when it came to the twist, there was no significant effect observed (Figure 22.b). Despite this, the coefficient of variation values did not exhibit the same level of consistency as the comparison between the results obtained from the different speeds in the tests (Figure 22.c).

Regarding the coefficient of variation values for force-elongation, similar to the H1 preparations previously, there are noticeable higher deviations in the maximum tensile force. This is particularly prominent in the FAST preparation, surpassing the allowed limit by over 100%. Additionally, in the elongation at break specification, FAST also exceeds the limit, although not as significantly as before (less than 25% above the limit). In terms of the other force-elongation specifications, once again, no clear trend towards the optimal preparation is observed.

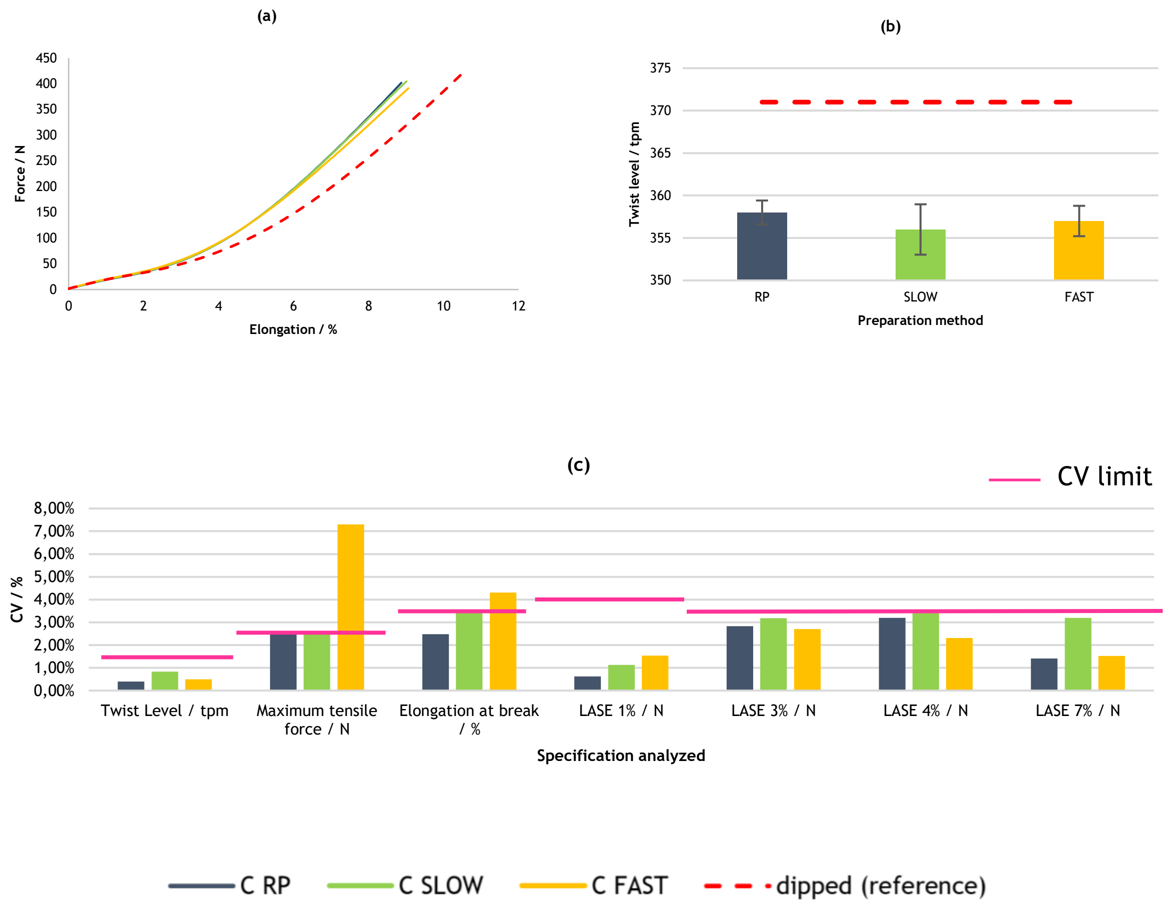


Figure 22. Test results for H1: (a) force-elongation test, (b) twist level test and (c) CV of speed of preparation test results.

SLOW preparation, however, has one major disadvantage compared to the practice itself. Here, the rubber tends to slip down the cord, requiring significantly higher tension to remove it, often damaging the cord and making it impossible to use the specimen. This drawback has only been observed for cap strip material.

The results obtained in H1 (Figure 22) exhibit a similarity to those in Figure 23 for N1. The different preparations have a minor influence on the force-elongation curves, while the twist level remains unaffected. Furthermore, unlike the previous material, all the coefficient of variation (CV) values in Figure 23.c. are below the established limits.

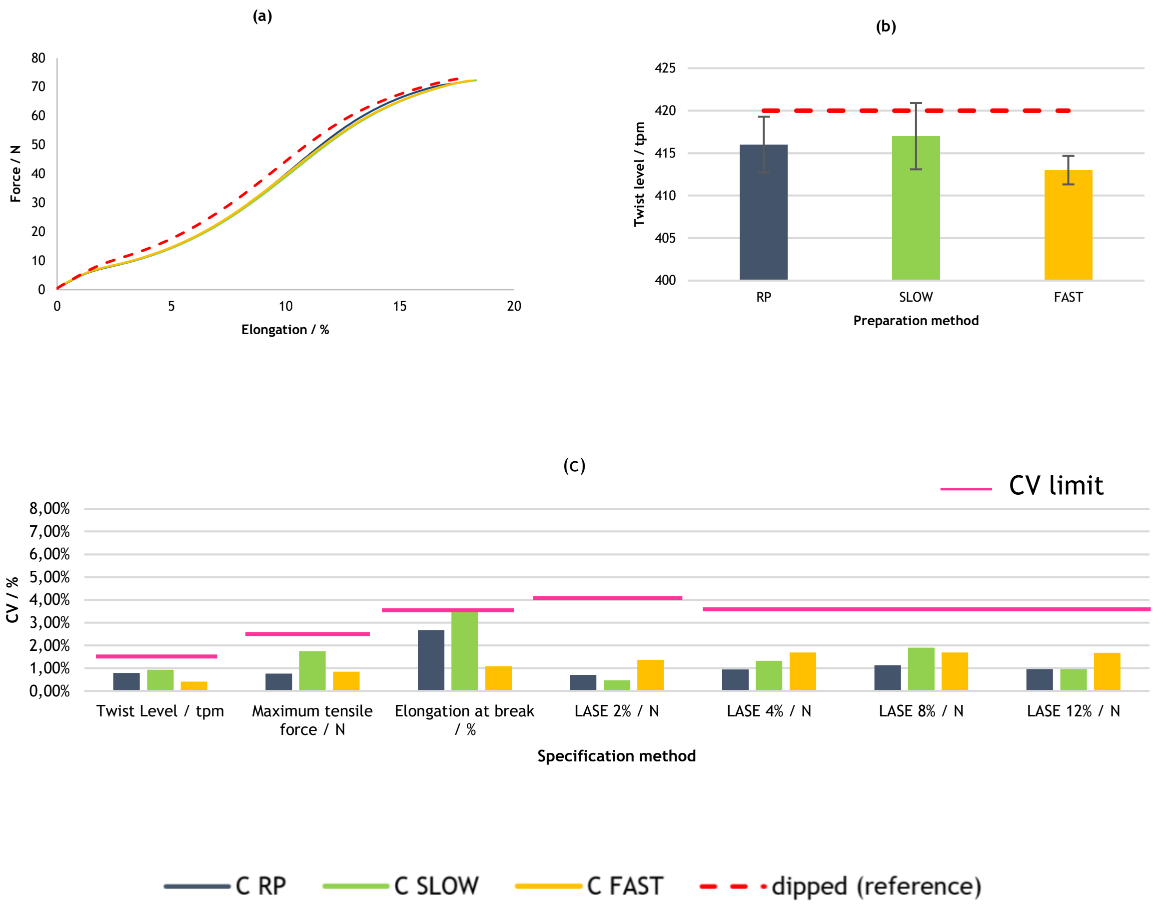


Figure 23. Test results for N1: (a) force-elongation test, (b) twist level test and (c) CV of speed of preparation test results.

Apart from that, there is no trend towards the best preparation. But in this case, all of them could be performed without affecting the results. Is should, however, be given a double attention to the force applied in the cord that occurs rubber slip, as it was mentioned before it happens often with cap strip material if it is prepared slowly.

In the case of P1 (Figure 24), it is a highly stable material when it comes to different preparations. It does not show significant effects on force-elongation, twist, or coefficient of variation (CV), ensuring consistent and repeatable results. However, SLOW and RP have shown more comparable results with regards to force-elongation specifications.

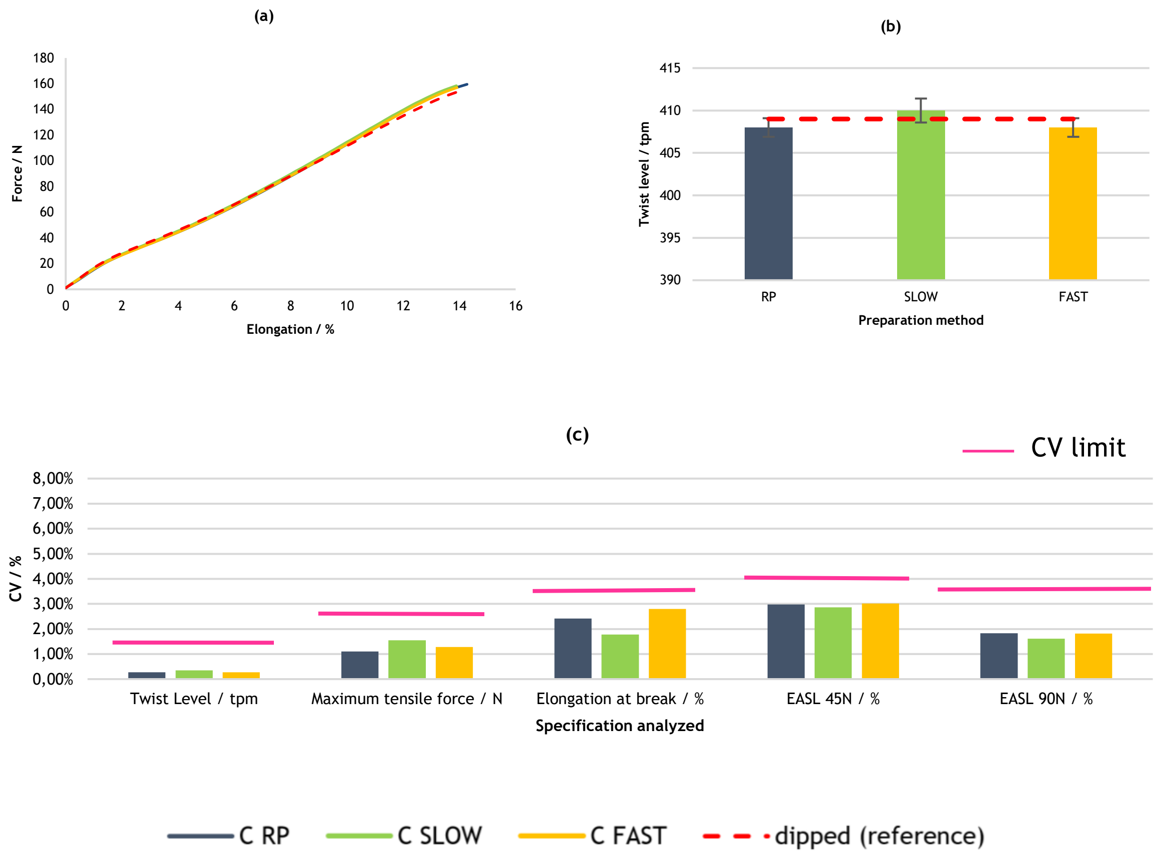


Figure 24. Test results for P1: (a) force-elongation test, (b) twist level test and (c) CV of speed of preparation test results.

Also, there is a trend towards slightly higher CV values observed in the specimens prepared using the FAST preparation. Nonetheless, any of the preparations can be performed without significantly interfering with the results.

Lastly, in the case of P2, there is a noticeable disparity in the force-elongation behavior between the RP, FAST and SLOW preparations. However, the values between the FAST and SLOW preparations are comparable, distinguishing them from the RP (Figure 25.a). As for the twist level, once again, no significant differences were observed (Figure 25.b).

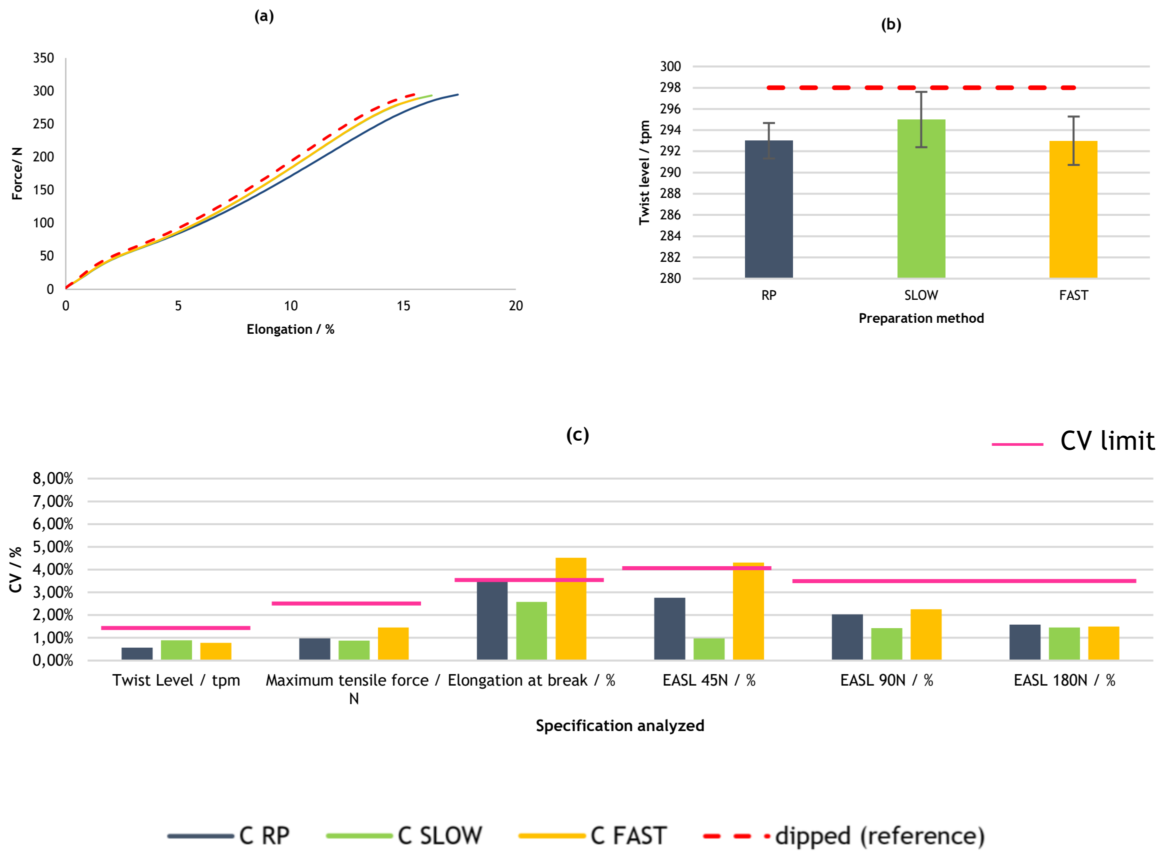


Figure 25. Test results for P2: (a) force-elongation test, (b) twist level test and (c) CV of speed of preparation test results.

Regarding the CV values, as observed previously in P1, there is a tendency for higher CV values in the FAST preparation. Additionally, in terms of elongation at break and EASL 45N, the CV values for the FAST preparation exceed the established limits.

Based on these findings, it is suggested to preferred performing the preparation slowly or in a random way, instead of in a fast pace.

4.1.4 Method of conditioning

As it was referred, the samples were analyzed by direct and conditioned testing. The comparison of the CV within samples in each test and for each material is shown in Appendix D. It is evident that conditioning the samples after pulling out the cord is crucial, as it leads to a strong trend of lower deviations once the samples have been relaxed through conditioning. This is particularly important because during the cord pull-out procedure, the cords are subjected to uncontrolled forces that can vary depending on the preparation, material, and operator. In direct testing, the cord is still influenced by these tensions, resulting in deviations in the sample results.

With that in mind, this portion of the study focused on analyzing how the samples could be effectively relaxed prior to testing. Without further ado, it was observed that for all tested materials, the conditioning methods did not have an impact on the force-elongation curves (Figure 26.a for H1, Figure 27.a for N1, Figure 28.a for P1, and Figure 29.a for P2), with only two exceptions in maximum tensile force for H1 and in EASL 45N for P1. The objective of testing the conditioning methods was not only to reduce CV values between samples but also to find a method that would best preserve the twist level during conditioning.

For H1 (Figure 26), the method that most preserved the twist level was L/N, while H method resulted in the highest twist loss, approximately 10 tpm, which is significantly low when compared to L/N (Figure 26.b). On the other hand, The H/N and L conditioning showed similar twist level.

Regarding its CV values for twist level tests, and analyzing the tests separately, it becomes apparent that conditioning the samples on a flat surface tends to result in lower deviations (Figure 26.c). However, note that all the tested conditioning methods have CV values that fall within the established limits, with L/N exhibiting the highest reproducibility.

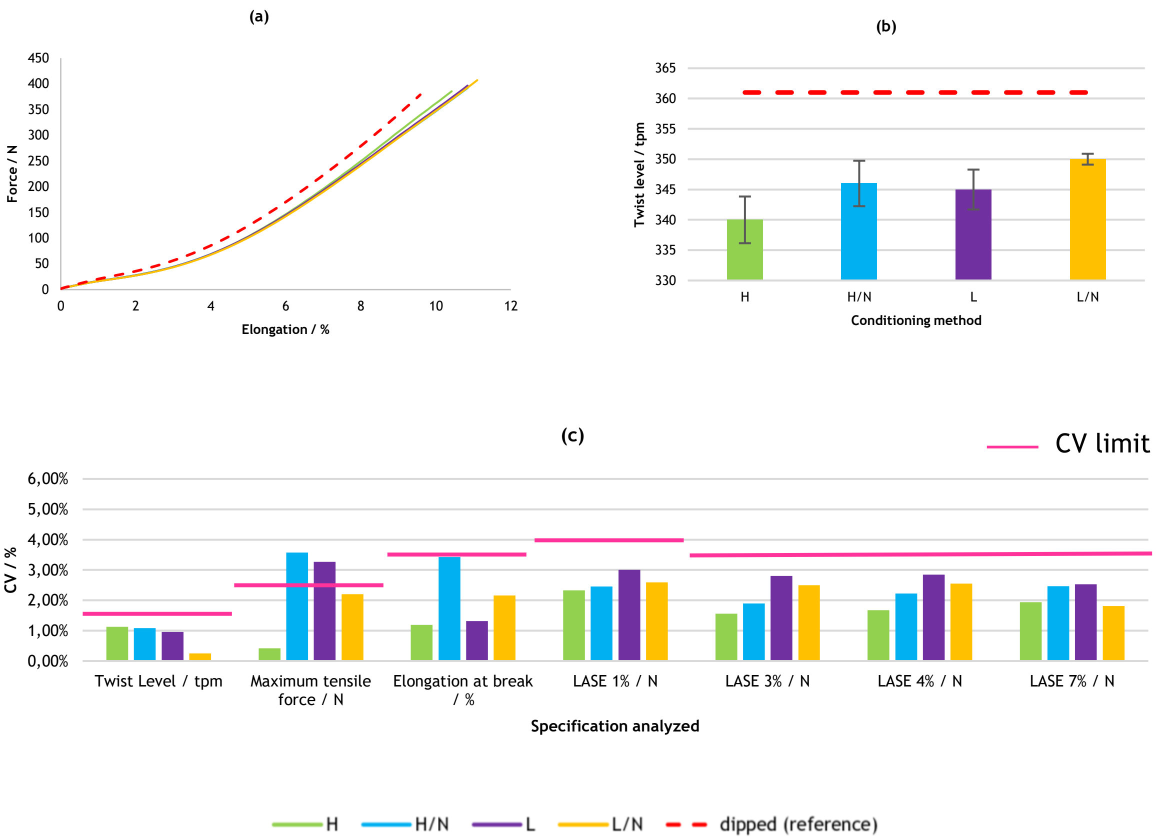


Figure 26. Test results for H1: (a) force-elongation test, (b) twist level test and (c) CV of method of conditioning test results.

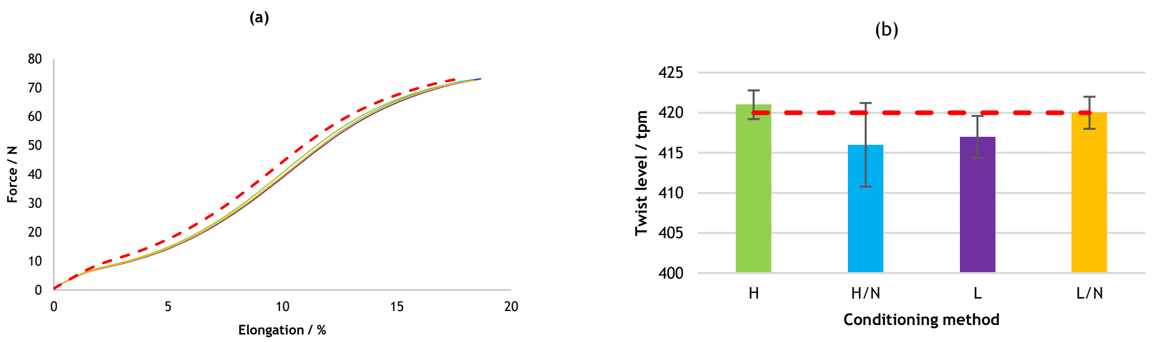
As for the force-elongation CV values, the H/N conditioning method displays the highest values for elongation at break and maximum tensile force, even surpassing the limit for the latter. On the other hand, H/N exhibits moderate behavior for the other specifications, consistently staying below the limits.

Higher CV values for the H and H/N conditioning methods can be expected. H, due to the cords being free and more prone to irregular twist loss, resulting in higher deviations in force-elongation results. H/N, on the other hand, causes the cords to wrap around themselves as if they were twisted again but in an irregular manner.

Considering both twist preservation and acceptable reproducibility, with CV values well below the established limits, L/N should be regarded as the most suitable conditioning method for this particular material. And the H method, should be avoided.

Regarding N1, the method that best preserved the twist level was H, followed by L/N (Figure 27). The H/N and L conditioning methods showed similar twist level.

Regarding its CV values, namely the twist tests CV values, there is no significant difference of CV among the different conditions. However, in terms of force-elongation CV values, in elongation at break, three methods of conditioning displayed above limits CV values: H, H/N and L. Only the L/H conditioning method was below the established limit for all the specifications. Additionally, there is a trend of decreasing CV values from left to right, indicating lower CV values for the laying conditioning method.



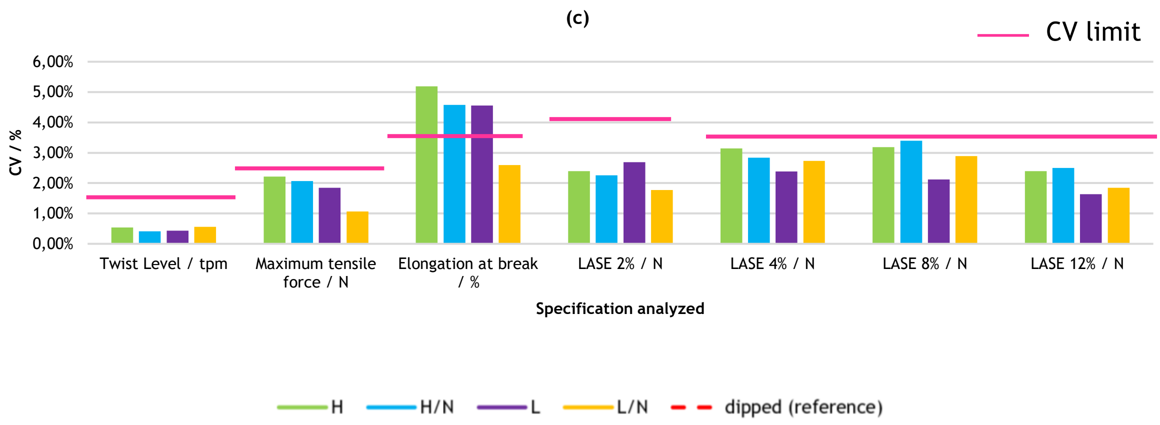


Figure 27. Test results for N1: (a) force-elongation test, (b) twist level test and (c) CV of method of conditioning test results.

Since L/N obtained the lower CV values in several specifications, and it is one of the methods of conditioning that twist level is most preserved, it is a highly recommended procedure.

As for P1 (Figure 28), being the most stable material during the trial of tests, no method of conditioning significantly affected the twist level (Figure 28.b) or even the CV values (Figure 28.c). The material presented low and similar CV values for both tests.

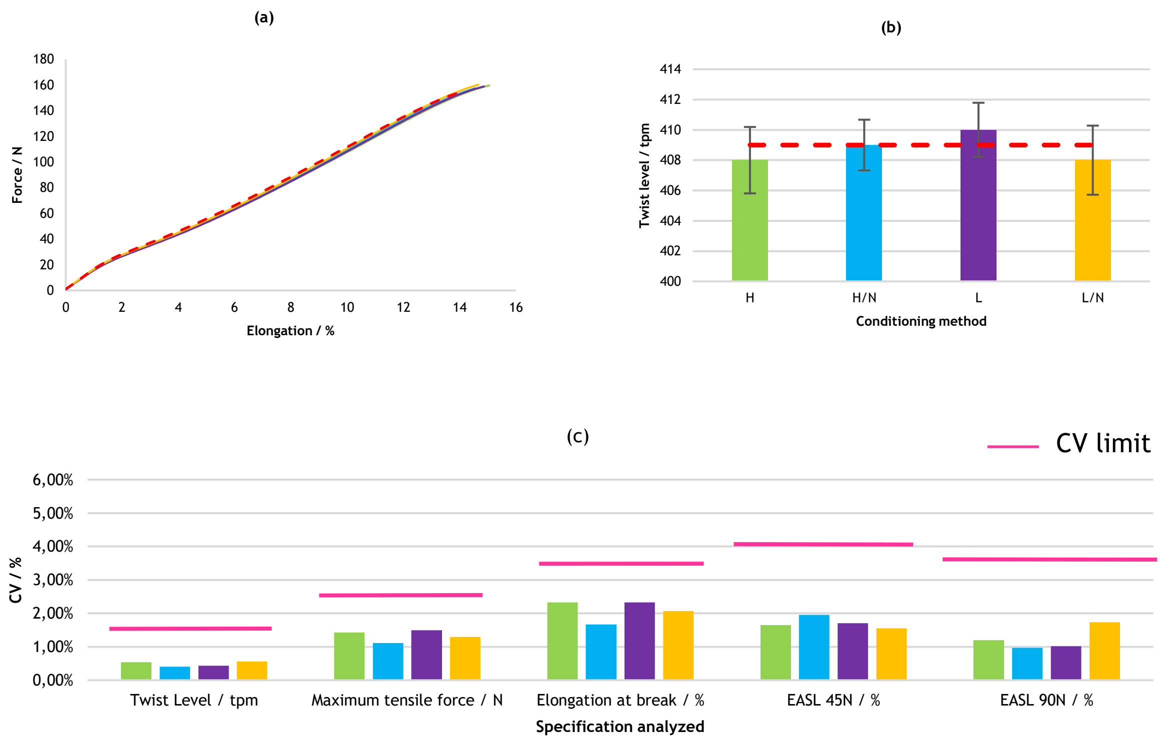


Figure 28. Test results for P1: (a) force-elongation test, (b) twist level test and (c) CV of method of conditioning test results.

That being said, there is no bias towards better or worse conditioning.

To conclude with P2, the method that most preserved the twist level was L/N, followed by H (Figure 29). Again, H/N and L conditioning showed similar twist level, however, significantly below when compared to L/N.

As for its twist level, the CV values are similar and quite under the established limits. In the force-elongation CV values, the only specification that exceeded the established limit was EASL 45N with the H conditioning method, while L/N had a CV value very close to the limit but still below it. For the other specifications, no trend behavior is observed. The CV values for different conditioning methods remained below the limits, with L/N having the lowest value in several specifications.

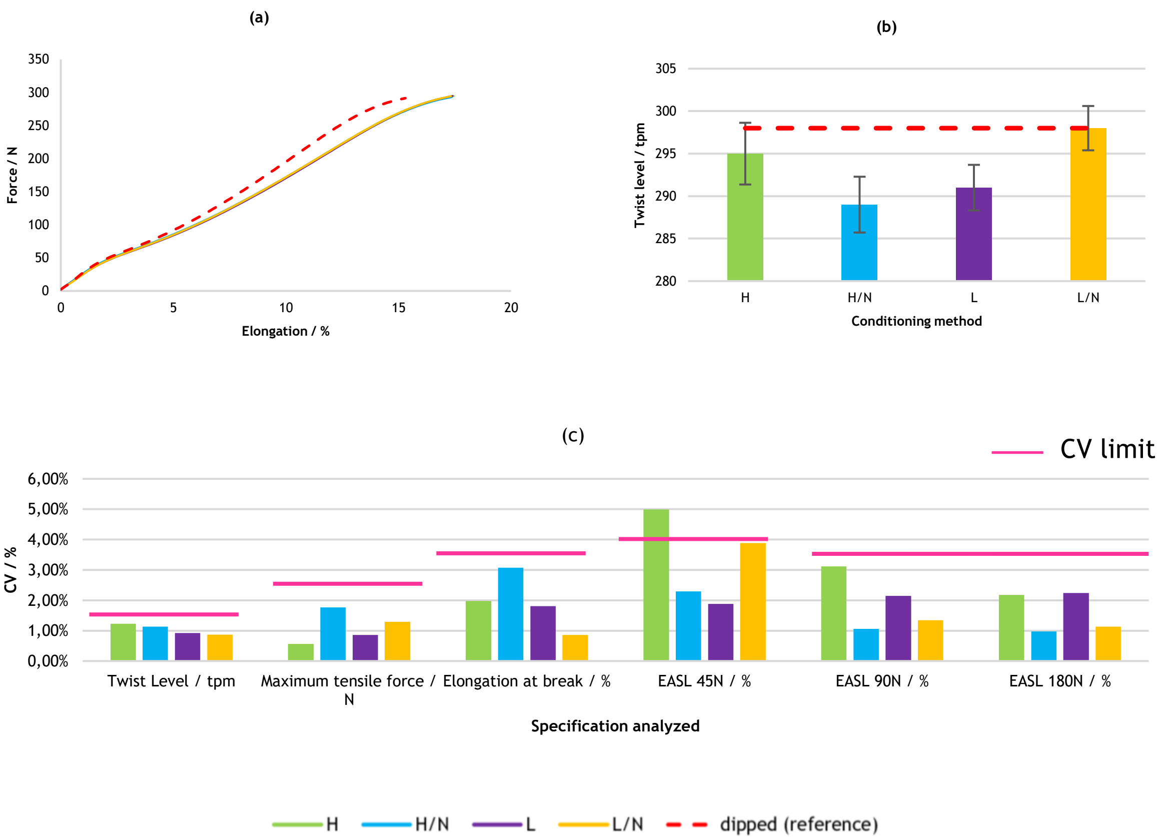


Figure 29. Test results for P2: (a) force-elongation test, (b) twist level test and (c) CV of method of conditioning test results.

In this situation, it is advisable to avoid using H and H/N conditioning method, due to the high CV values and the low twist level, respectively.

4.2 Cross-check testing

The second part of the study involved a coordinated cross-check testing between five different laboratories belonging to Continental. In this phase, only H1 and N2 were tested.

The concept of this was to investigate the current procedures, handling, and testing methods used in different laboratories. Additional tests were conducted to compare the results obtained. As the material had the same history and originated from the same process, the impact of the manufacturing process on the samples could be disregarded. This allowed the results to be attributed solely to the handling, conditioning, and testing procedures. The results obtained from the tests conducted in other laboratories were compared with those from Laboratory 1 – laboratory in which all the tests of this work were performed.

The test results were evaluated based on the specification range of values determined by the company, represented by the red error bars. This implies that the results from the other laboratories should not exceed this range, otherwise it indicated a potential flaw in the test or an influential input factor, when compared to Laboratory 1. The dark grey bars represent the deviation within the samples for the respective laboratory.

The results of both twist level and force-elongation tests for the dipped material are presented in Appendix E. The force-elongation test results for N2 are depicted in Figure 30, while those for H1 are shown in Figure 31. It should be noted that Laboratory 4 did not perform the twist level test.

For N2, it can be observed that all the laboratories achieved similar values in force-elongation tests (Figure 30.a and Figure 30.b), allowing a comparison of both the direct and conditioned results. The range for the smaller specifications might be difficult to visualize, but all the results fall within the acceptable limits, as detailed in Appendix E.

The same similarities have been observed for twist level (Figure 30.c and Figure 30.d). There is a tendency for slightly lower twist level values in the results from the other laboratories, although the difference is not substantial.

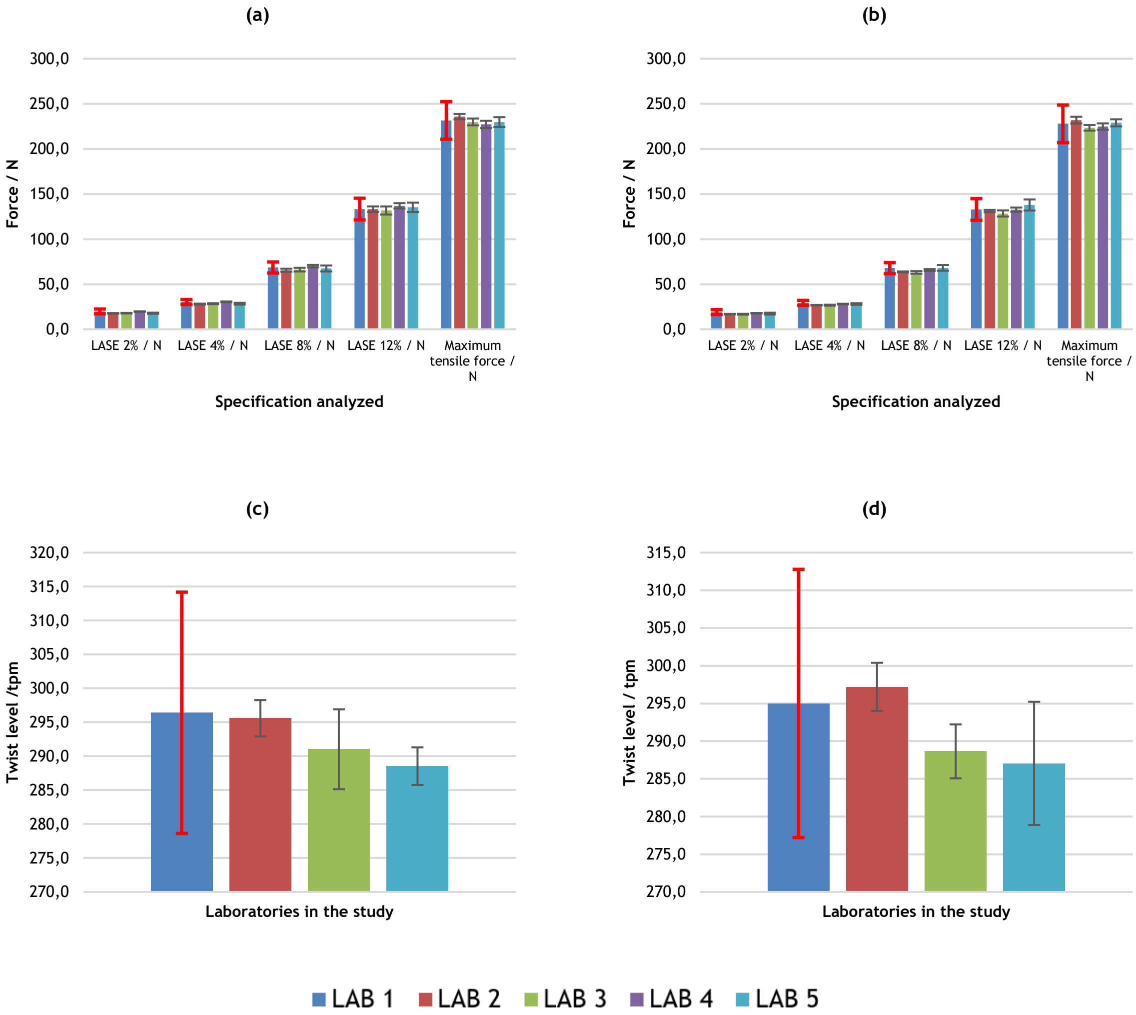


Figure 30. Force-elongation and twist level test results for N2 rubberized, direct (a and c) and conditioned (b and d), respectively.

Regarding H1, there were inconsistencies observed in the results (Figure 31). This discrepancy was particularly noticeable for higher specifications such as LASE 7% and maximum tensile force, both for direct and conditioned samples (Figure 31.a and Figure 31.b). These results were not entirely in line with expectations, as the objective was to achieve consistent and accurate results across different laboratories. However, it was noted that H1 is a highly sensitive material and prone to deviations even with a single operator. Therefore, when multiple operators are involved, some level of deviation can be anticipated. However, the extent of deviation observed was more significant than anticipated.

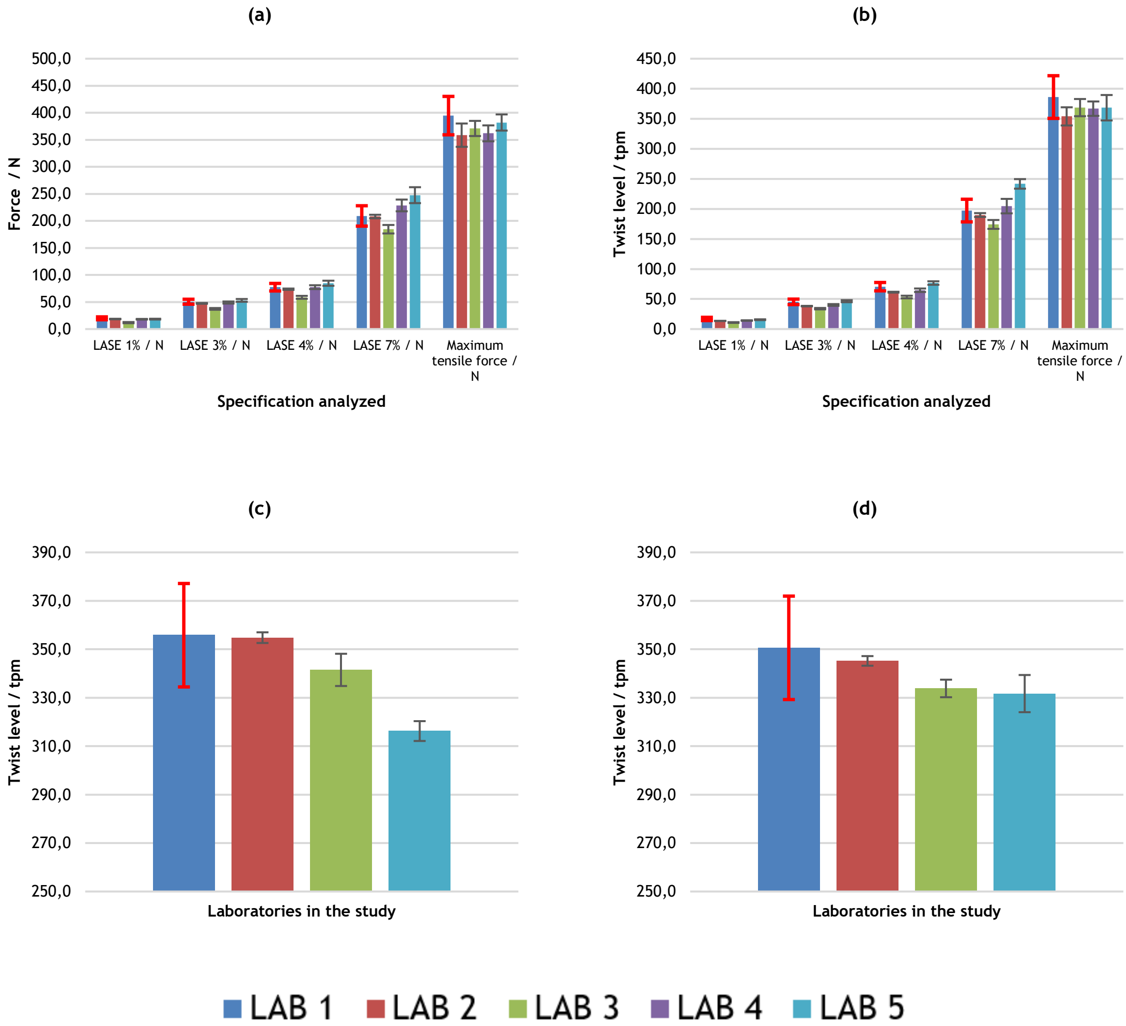


Figure 31. Force-elongation and twist level test results for H1 rubberized, direct (a and c) and conditioned (b and d), respectively.

There are several hypotheses regarding the root causes that could have influenced the results, particularly concerning the material handling, conditioning, and equipment used. The mention of equipment is relevant because the test clamps employed for force-elongation tests varied for the materials tested, and since the results for N2 were acceptable, this could be a contributing factor.

Regarding the twist level (Figure 31.c and Figure 31.d), when tested directly, the samples exhibited higher variations in twist compared to when tested in conditioned state. In the first case, the difference between the highest measured twist level (Lab 1) and the lowest (Lab 5) is approximately 40 tpm, which is quite significant. For the conditioned results, although there is a difference between the laboratories, the values are within the acceptable range. Once again, Lab 5 displayed lower twist level values. The same root cause hypothesis considered for force-elongation tests may also be applicable in this scenario.

Lastly, upon analyzing the deviations within N2 specimens for each test and laboratory, similar trends can be observed (Figure 32). As expected, conditioned samples exhibited lower deviations. However, only Lab 3 (Figure 32.c) and Lab 5 (Figure 32.e) presented deviation values surpassing the established limits.

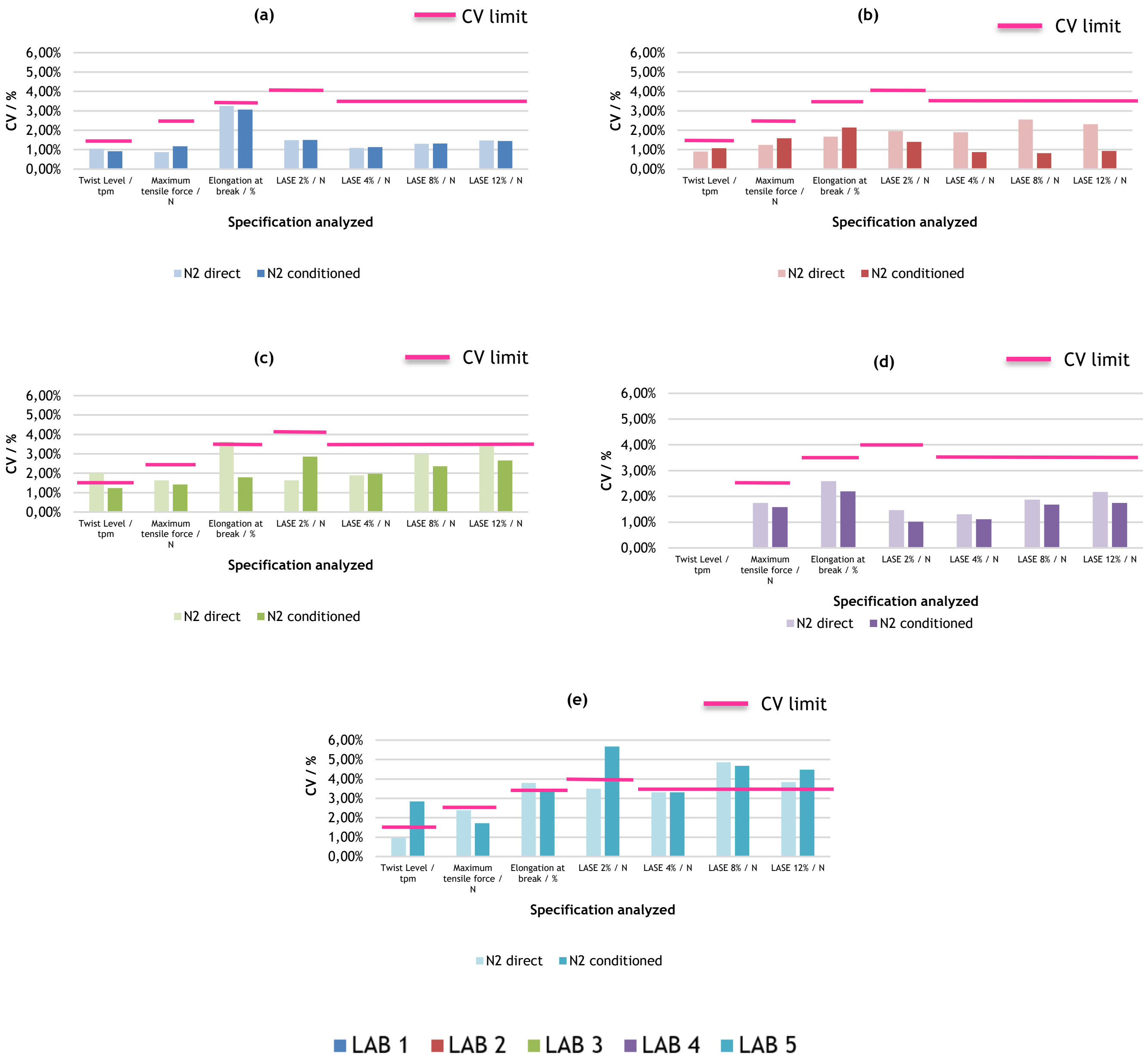


Figure 32. Coefficient of variation of N2 for each laboratory (direct and conditioned testing).

Regarding H1 (Figure 33), notably higher deviations were observed. All laboratories exhibited coefficient of variation (CV) values for maximum tensile force that exceeded the established limits. Lab 2 (Figure 33.b) had the highest value, which was twice the allowed limit, followed by Lab 5 (Figure 33.e). A similar situation occurred for Lab 3 in terms of elongation at break, where the values exceeded more than double the permissible limit (Figure 33.c).

Furthermore, it is worth noting that the two laboratories (Lab 3 and Lab 5) with the lowest twist levels for both materials (Figure 32 and Figure 33) generally displayed higher deviations compared to the other laboratories. As previously mentioned, the irregular twist loss might contribute to increased deviations in the test results.

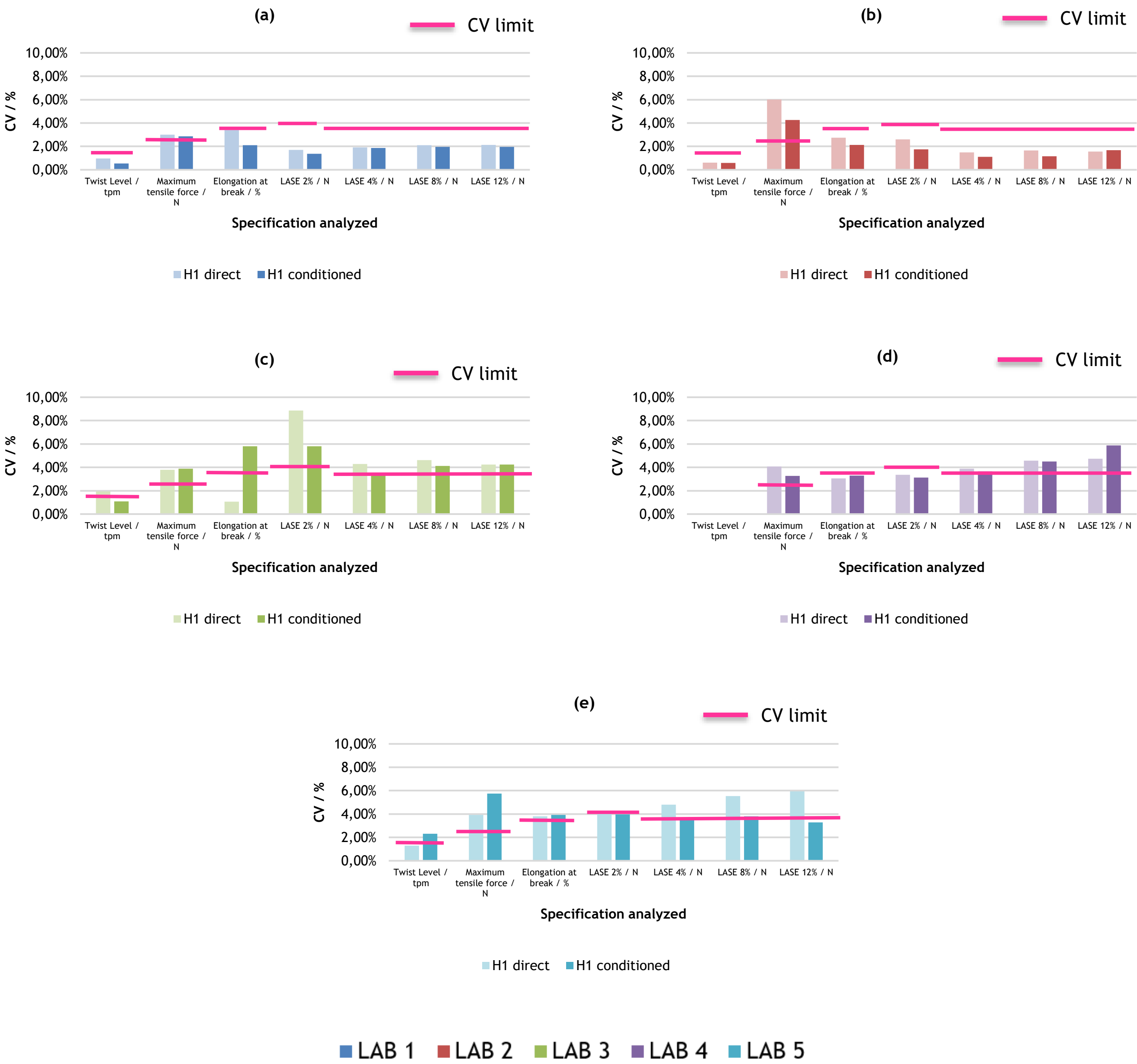


Figure 33. Coefficient of variation of H1 for each laboratory (direct and conditioned testing).

To comprehend the results and reach meaningful conclusions, it is necessary to replicate the tests.

5 Conclusion

This work's overall aim was to study the impact of different sample preparation and conditioning procedures on tire rubberized cord, particularly with regard to inter-sample comparability and reliability of results. The cord materials studied were hybrid (H1, aramid + nylon6.6), nylon 6.6 (N1 and N2) and polyester (P1 and P2).

Regarding the manner of preparation, the material that showed more variability in force-elongation tests was P2, followed by N1. The most affected in twist was H1. It was also observed that the preparation with cotton gloves (CotG) resulted in twist loss across all materials. Therefore, it is advisable to avoid using this preparation method. In terms of coefficient of variation (CV), the preparation with sweaty hands (SWEATY) and with hand-cream (CREAM) consistently exhibited higher CV values. On the other hand, the preparations with clean and dry hands (CLEAN) or with rubber gloves (RubG) generally yielded more reliable and acceptable results. It is worth noting that, for optimal performance, it is crucial to ensure that the gloves used during the preparation process are of the appropriate size for the operator.

From the position and speed of preparation it was possible to observe reproducibility among cap strip and among calendered materials. Coincidently for cap strip materials, the pulling only cord preparation (PCP) consistently resulted in the highest twist loss during preparation. On the other hand, fixed-end preparation (FEP) tended to preserve the twist level well and facilitated the pull-out of cords, although it showed some high CV values for H1. If the tendency of high CV values persists, it is recommended to investigate and analyze the root cause further. In this context, random preparation (RP) and difficult preparation (DP) are strongly recommended for cap strip materials.

For calendered materials, the twist level was not significantly affected by the preparation method. However, P2 showed the most pronounced effects on force-elongation. Interestingly, P1 was minimally influenced by the position of preparation. It is noteworthy that both materials, despite having different constructions, displayed a similar behavior in force-elongation when prepared using easy preparation (EP), showing lower initial modulus and higher maximum tensile force. The remaining preparations can be applied to calendered materials, with RP, DP, and PCP being more suitable. However, it is essential to perform PCP slowly, as indicated by the findings on the speed of preparation.

The speeds used for the pull-out barely affected the behavior of the specimens for force-elongation and twist tests. However, for certain materials, variations were noticed among specimens with high coefficient of variation (CV) values, particularly for H1 and P2. In the case of cap strip materials, no specific trend was observed. It is worth noting that excessively fast preparation could potentially affect the maximum tensile force (H1) and elongation at break

(H1 and N1). Conversely, overly slow preparation can lead to the rubber slipping down the rope more quickly, implying the discard of the specimen. Therefore, RP is the most recommended method. As for calendered materials, no significant differences were observed. However, both tested materials exhibited a tendency towards lower CV values with slower preparation, thus emphasizing the importance of a slow preparation approach. Again, RP is the recommended procedure.

Regarding the conditioning, the methods tested did not affect the force-elongation curves. In a general way, laying with a knot (L/N) was one of the methods that most preserved twist and presented low CV values, thus the method most advised to be applied. By contrast, hanging method (H) should be avoided as it presented high CV values for some of the tested materials.

Finally in the cross-check testing, it could be observed that even materials coming from the same production process, the handling, conditioning, and testing have an impact onto the results. To comprehend the results and reach meaningful conclusions, it is necessary to replicate the tests. However, some preliminary observations can be made based on the available data. H1 appears to be a highly sensitive material, particularly when it comes to handling, preparation, conditioning, and, notably, the rubberizing process. These factors contribute to the occurrence of higher deviations in the results. On the other hand, it is possible to infer that any potential impacts of the preparation process on N2 were counteracted by the conditioning phase. This deduction is supported by the similarity observed in the results for N2, where deviations remained within the established limits. Therefore, conditioning seems to have reversed or mitigated the effects that the preparation process may have initially had on N2.

In summary, the preparation procedure is advisable to be done with clean, dry hands, with the possibility of using rubber gloves if an individual has naturally sweaty hands or encounters any other issues that justify the use of gloves during preparation. As for the cord-pulling position, it is recommended to do it randomly (RP) - considering that in this preparation the cord chosen to pull out would come out easily and without much effort and tension. For cap strip materials, pulling only the rubber on the edges can facilitate the pull out (DP). For calendered materials, pull the cord from the edge of the strip (DP) and pulling only the cord out of rubber compound (PCP) also makes the procedure easier.

6 Assessment of the work done

6.1 Objectives Achieved

This work targeted study the impact of preparation and conditioning procedures on the tire rubberized cord, especially the impact of the preparation reflected in the quality test results. It was also performed a cross-check testing to verify the reproducibility of a test result by comparing it with test results from other laboratories. The goal after this study was to develop a new samples' preparation procedure that can guarantee reliable and reproducible results in all laboratories.

Through the conducted tests, it was evident that both preparation and conditioning have a significant impact on the test results. Moreover, it was observed that each material has its own optimal and most effective preparation method. Nevertheless, it was still possible to develop a clear, visually understandable preparation procedure that can accommodate various materials and can ensure greater repeatability in results and inter-laboratory reproducibility.

6.2 Final Assessment

Being able to work on my thesis in an international environment and with so much autonomy and freedom to be curious, developed in me skills that made me grow personally and professionally and I am sure I will carry with me for life. Additionally, the opportunity to apply the knowledge gained from my studies has provided me with practical learning experiences and the chance to exchange knowledge and insights, which has been truly rewarding. Given that, had the opportunity to put into practice a great deal of what I learned in my academic career and acquire more knowledge, mainly related to soft skills. We were guided to be independent, and it was challenging to assimilate a large amount of new information quickly and make decisions based on the numerous options we were presented with. As a result, I was delighted to see the value of my work for Continental and to feel that my efforts contributed to the advancement of textile reinforcement characterization techniques. Given that, it is hopeful that the developed preparation procedure can yield more reliable, consistent and comparable results within Continental group.

6.3 Institutional acknowledgements

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Appendix A - Tire reinforcement terminology

Table A 1. Tire reinforcement terminology (Adapted from [5, 6]).

Terminology	Description
Fiber	Linear macromolecules orientated along the length of the fiber axis.
Filament	The smallest continuous element of a tire cord.
Yarn/Strand	An assembly of filaments usually gathered directly from the creel, twisted lightly, and rolled up on beams for further processing.
Cord	A twisted or formed structure composed of two or more yarns.
Warp	Cords in a fabric that run lengthwise.
Pick/Filling	Light threads that run at right angles to the warp that serves to hold the fabric together (weft).
Tex	Cord weight expressed in grams per 1000 m.
Denier	Cord weight expressed in grams per 9000 m.
Decitex	The linear density in SI units (partially), weight in grams per 10000 m. Can be written as dtex.
Construction	The way of identifying the specification of a cord, expressed by the linear density and number of yarns (ex.: 1400dtex x2).
Rivet	Distance between cords in a fabric; a high rivet count typically describes a fabric with a low EPDM.
EPDM	Ends of cord per dm width of fabric.
LASE	Load applied to a cord for a specified elongation (Load At Specified Elongation).
EASL	Elongation when a specified force is applied to the cord (Elongation At Specific Load).
Twist	The number of 360° turns per unit length of yarn or cord. Twist level can be expressed by twist per meter (tpm) or twist per inch (tpi).
Twist direction	The direction of twist can be either clockwise ("S" twist) or counterclockwise ("Z" twist).
Strength	The tensile load required to rupture a cord at a given rate of extension. Tire cord strength is reported in Newtons or pounds.
Tenacity	Cord strength is frequently expressed in grams per denier or centinewtons per decitex.

Appendix B - Demonstration of samples' preparation

This appendix illustrates how the cords are inside the rubber matrix (Figure B 1 and Figure B 2) and how some of the samples' preparation and conditioning methods were done. The yellow arrows represent the direction in which the tension to pull out the cord or to tear the rubber is applied (Table B 1).

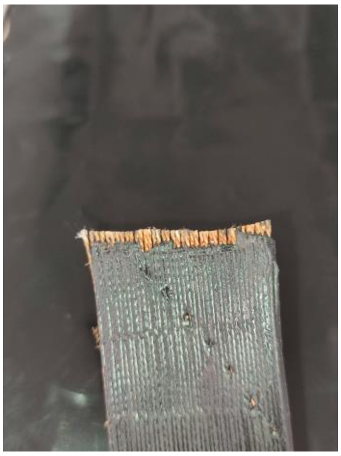
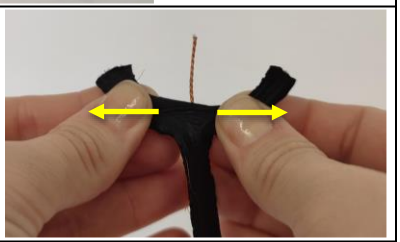


Figure B 1. Example of textile cord after calendering process.

Figure B 2. Example of textile cord after cap strip line process.

Table B 1. Demonstration of samples' preparation and conditioning methods.

CODE	Demonstration	
RubG		
CotG		
EP		

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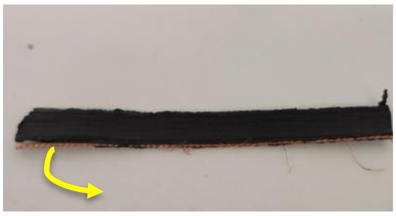
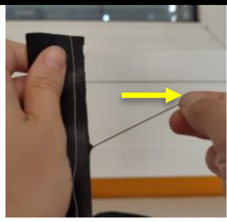
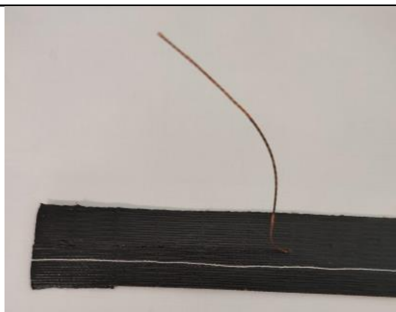
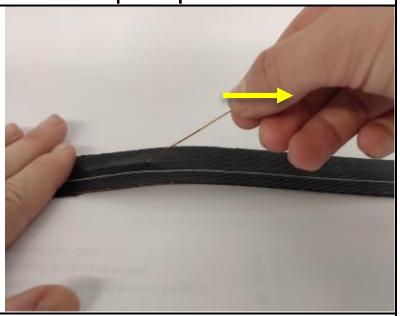
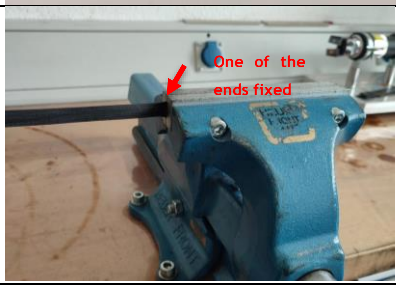
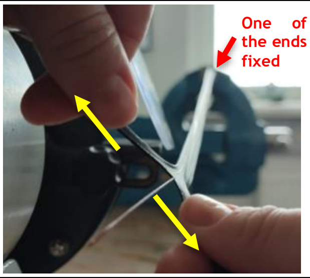
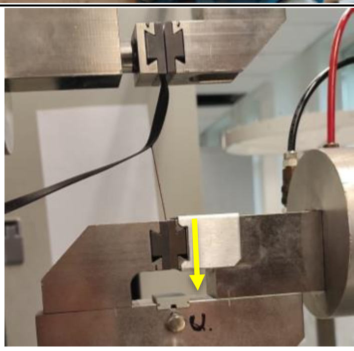
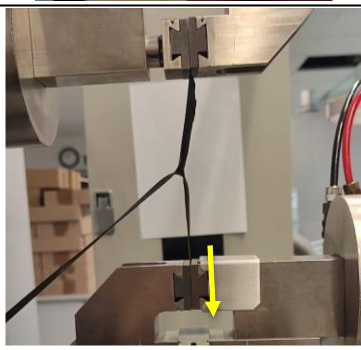
<tr><td>DP</td><td></td><td>
Calendered material


Cap strip material</td></tr>
<tr><td>PCP</td><td></td><td></td></tr>
<tr><td>FEP</td><td>
</td><td>
</td></tr>
<tr><td>SLOW</td><td rowspan="2">
Calendered material</td><td rowspan="2">
Cap strip material</td></tr>
<tr><td>FAST</td></tr>
<tr><td>H</td><td colspan="2" rowspan="2"></td></tr>
<tr><td>H/N</td></tr>
</table>

<table>
<tr><th>CODE</th><th colspan="2">Demonstration</th></tr>
<tr><td>L</td><td rowspan="2"></td><td rowspan="2"></td></tr>
<tr><td>L/N</td></tr>
</table>

Appendix C - Analysis of force-elongation results

In this appendix is presented the force-elongation test results in a more detailed mode, namely, the values obtained in each specification and their SD.

Manner of preparation

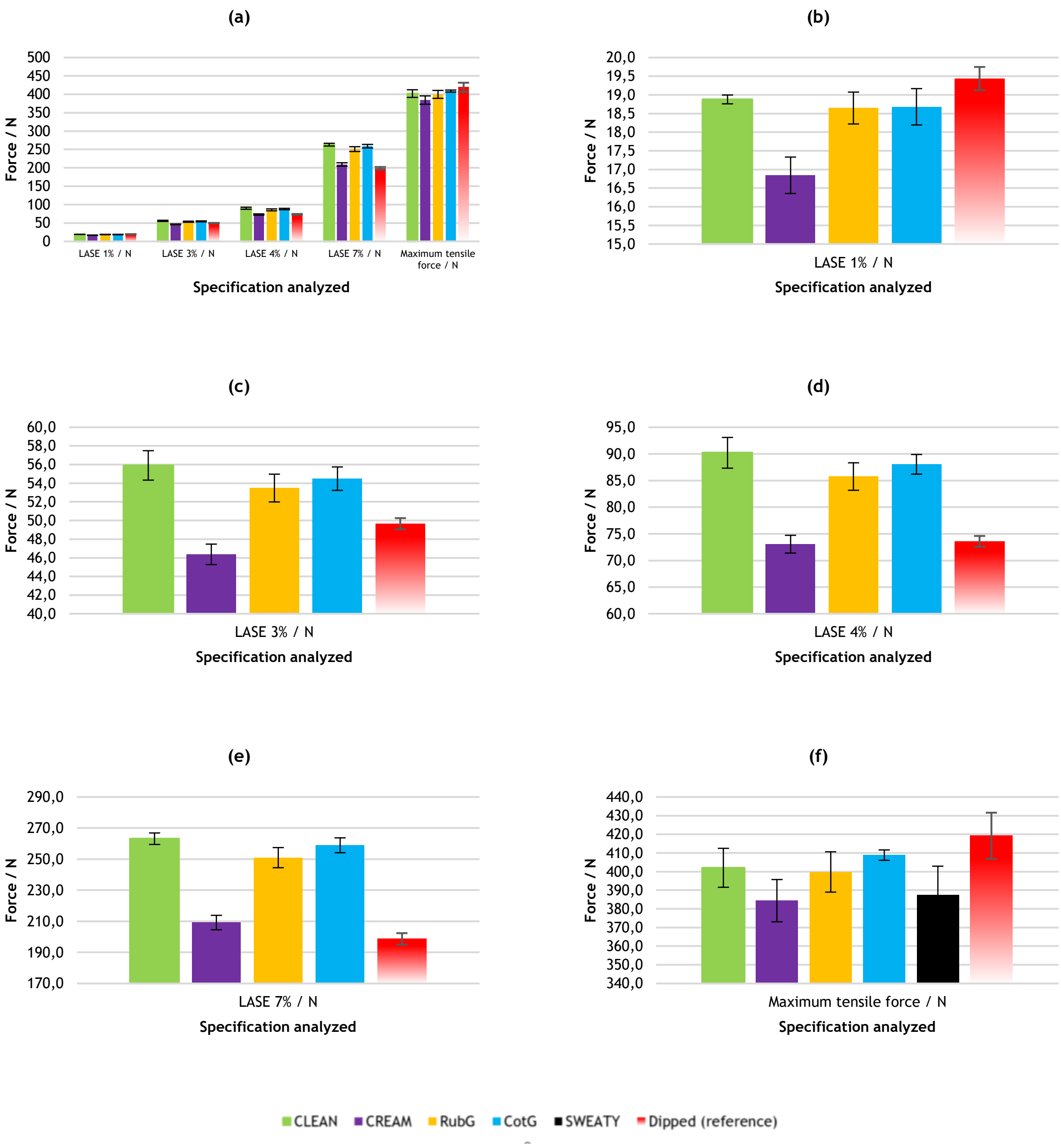


Figure C 1. H1 Force-elongation test results detailed in each specification and their SD.

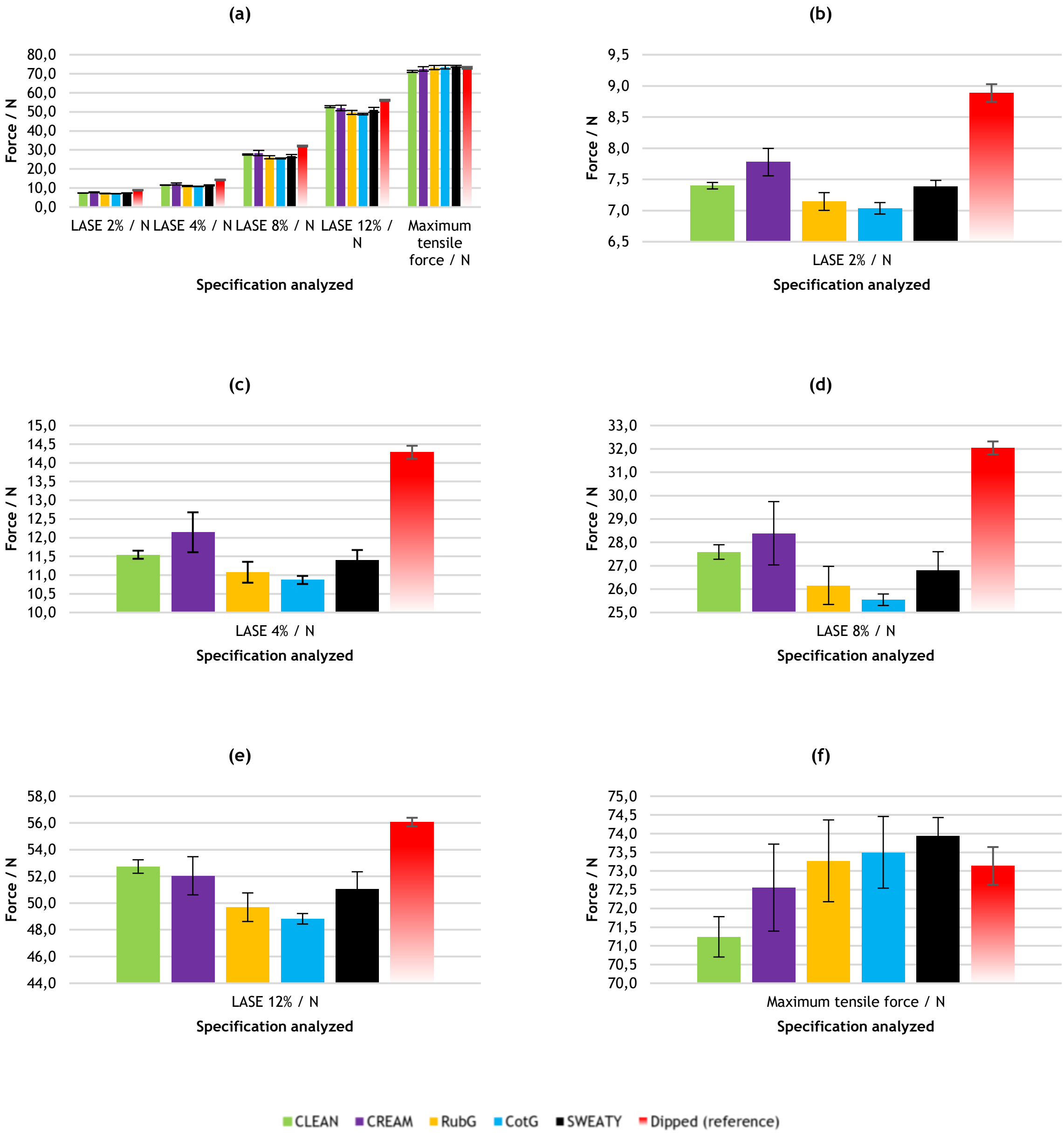
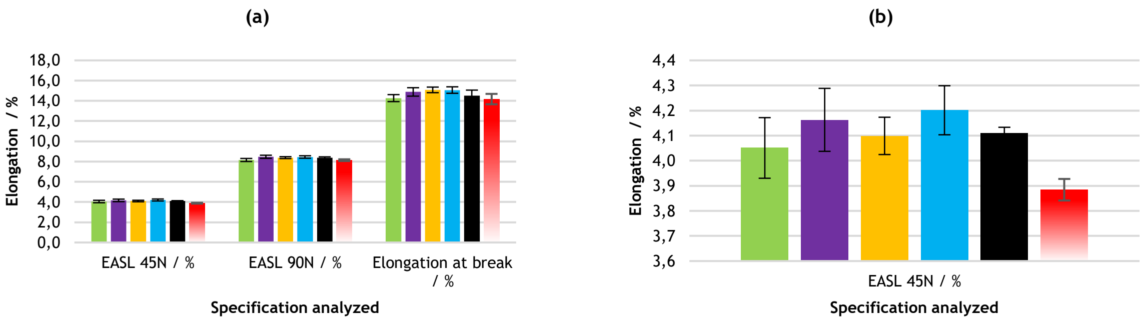


Figure C 2. N1 Force-elongation test results detailed in each specification and their SD.



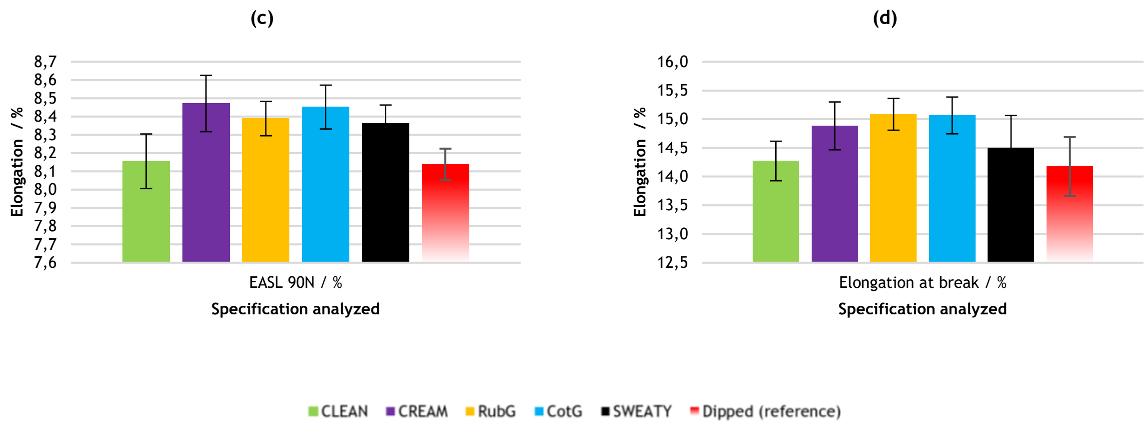


Figure C 3. P1 Force-elongation test results detailed in each specification and their SD.

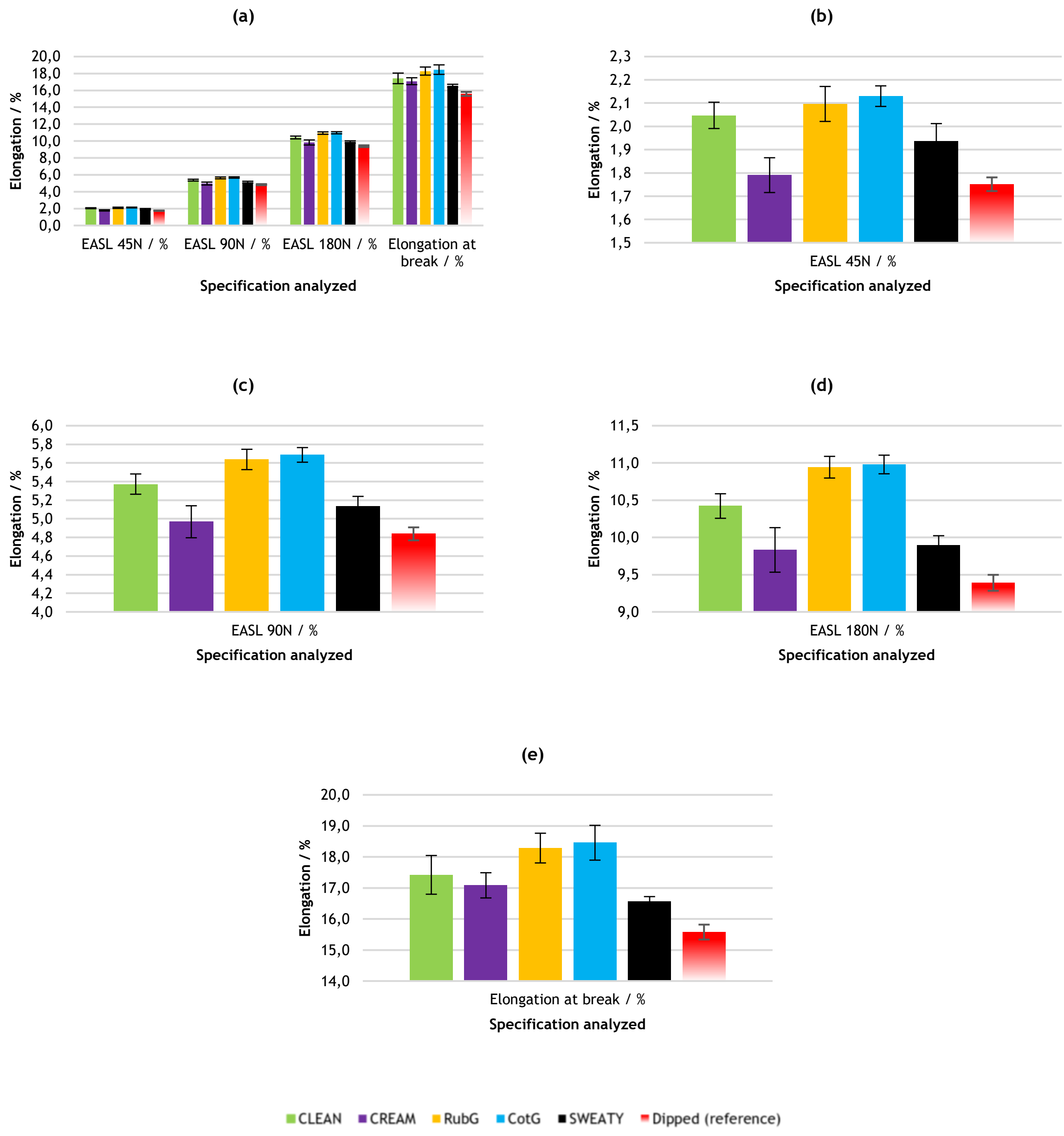


Figure C 4. P2 Force-elongation test results detailed in each specification and their SD.

Position of preparation

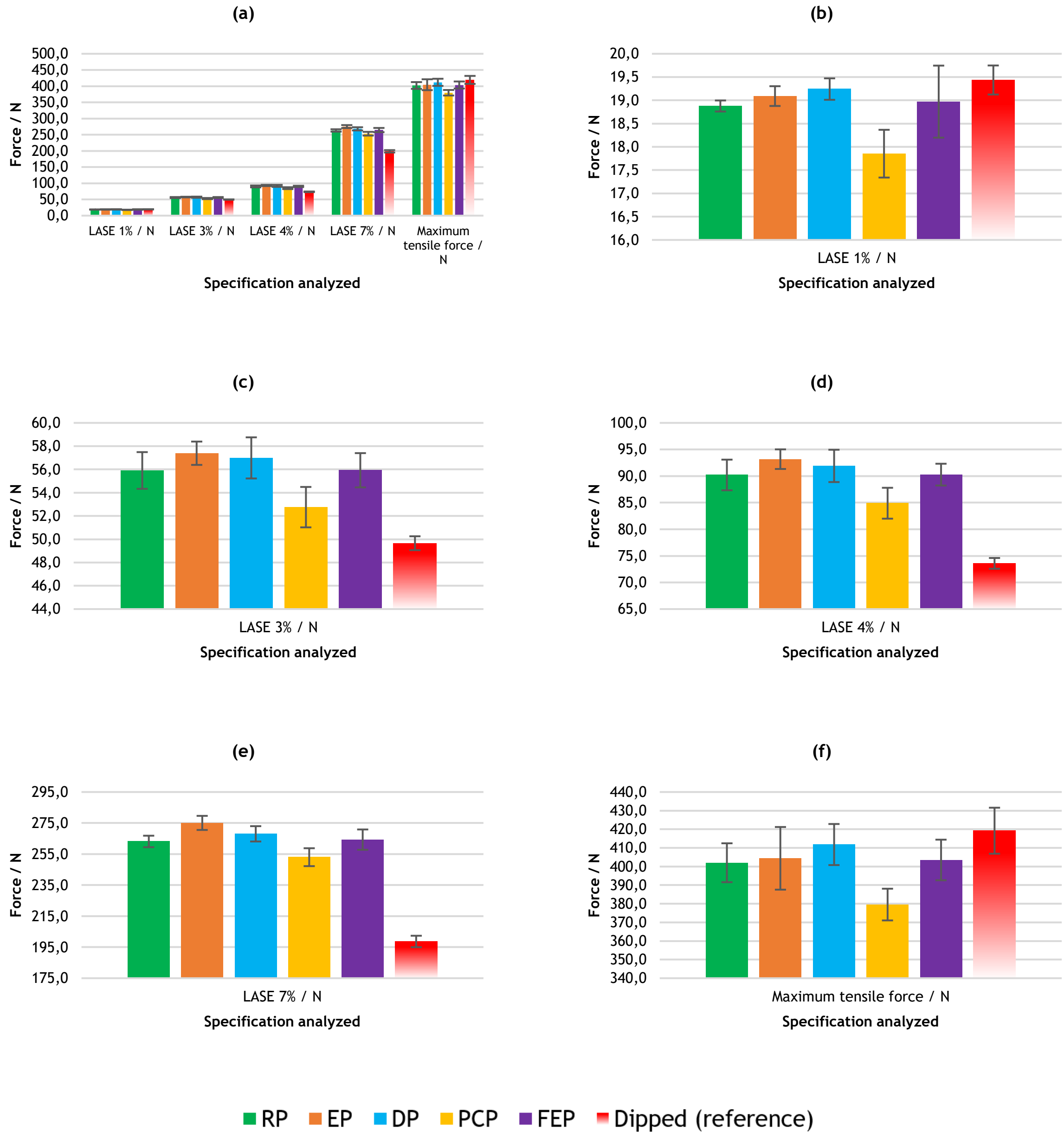
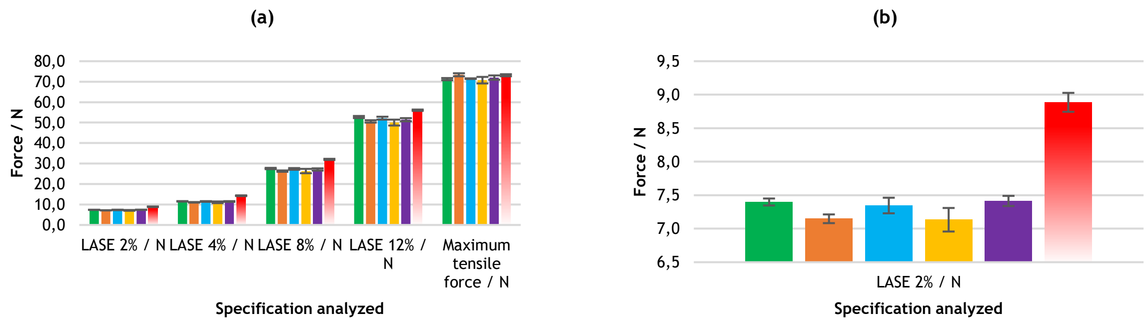


Figure C 5. H1. Force-elongation test results detailed in each specification and their SD.



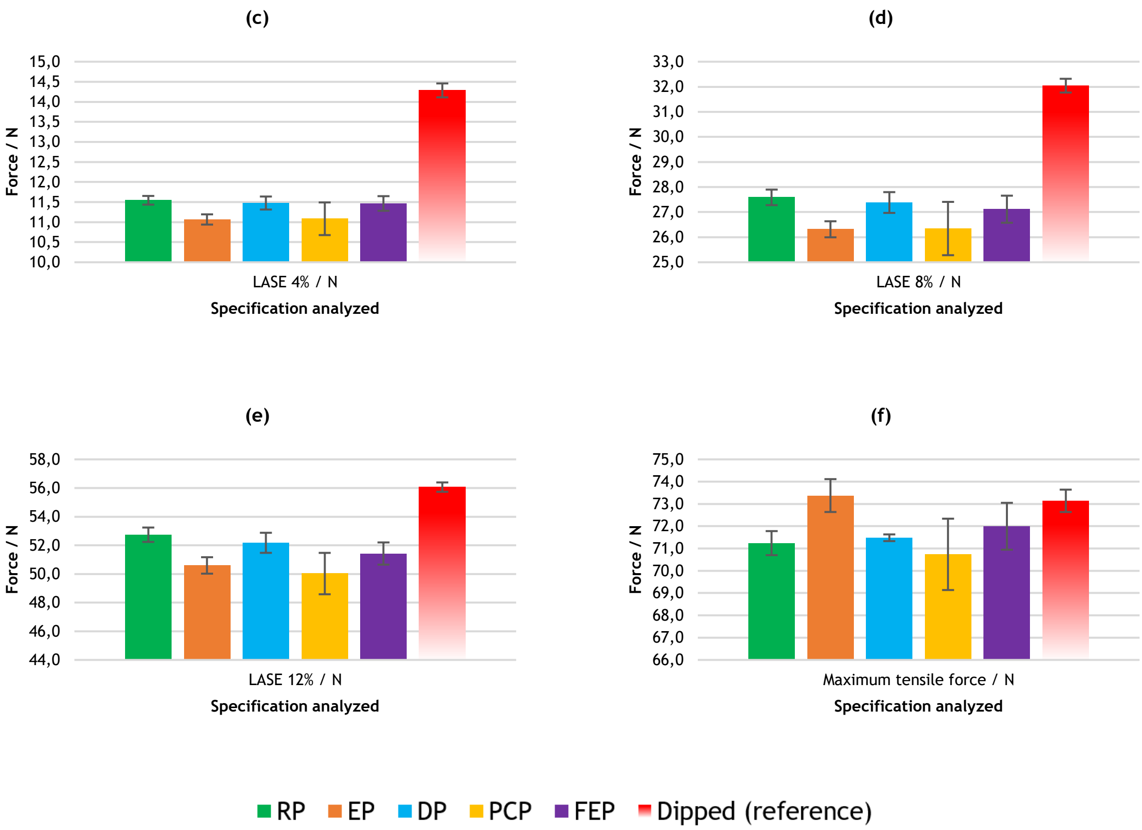


Figure C 6. N1 Force-elongation test results detailed in each specification and their SD.

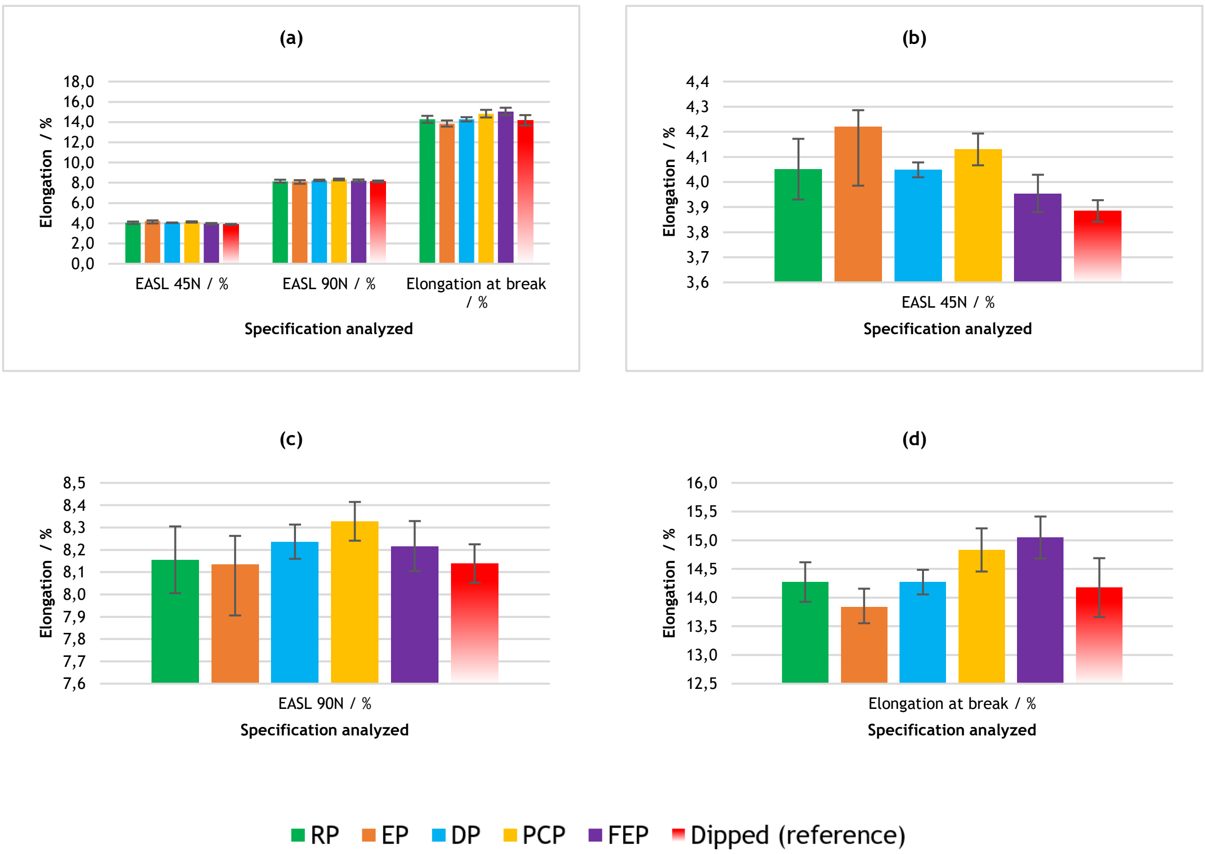


Figure C 7. P1 Force-elongation test results detailed in each specification and their SD.

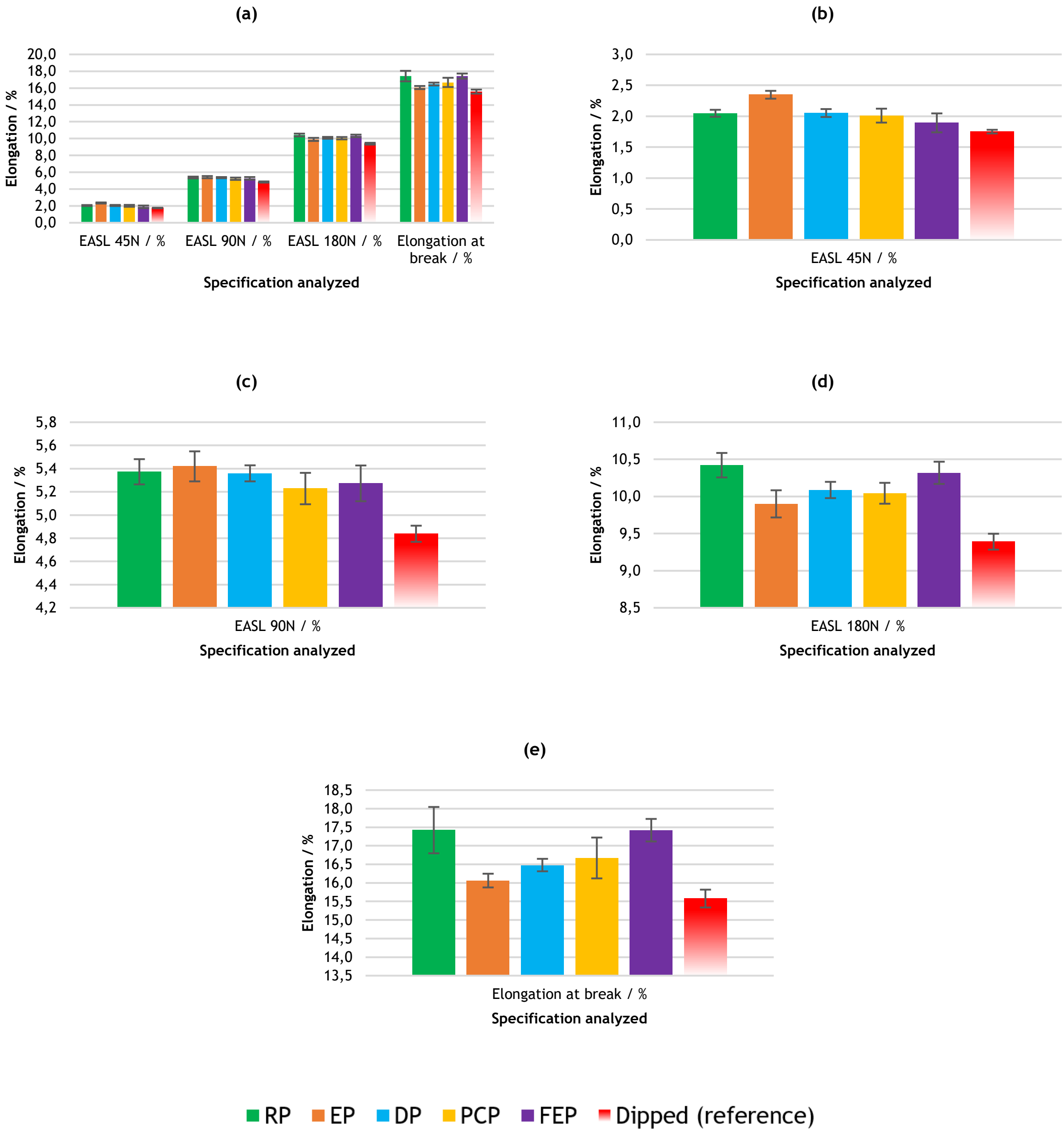


Figure C 8. P2 Force-elongation test results detailed in each specification and their SD.

Speed of preparation

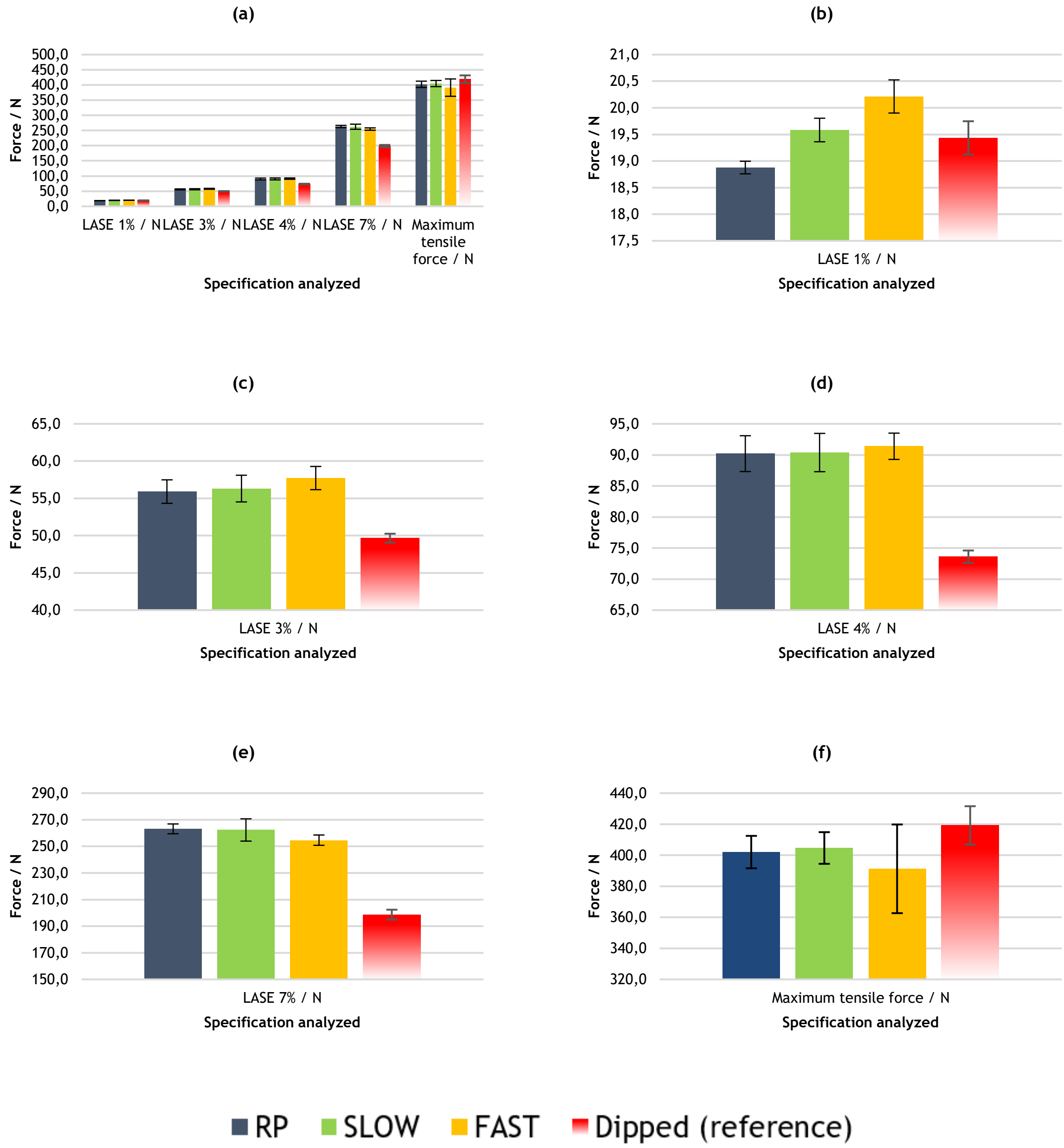
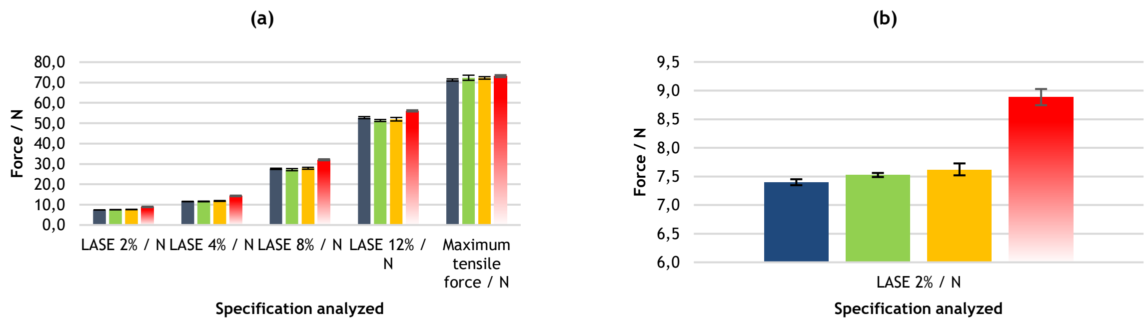


Figure C 9. H1 Force-elongation test results detailed in each specification and their SD.



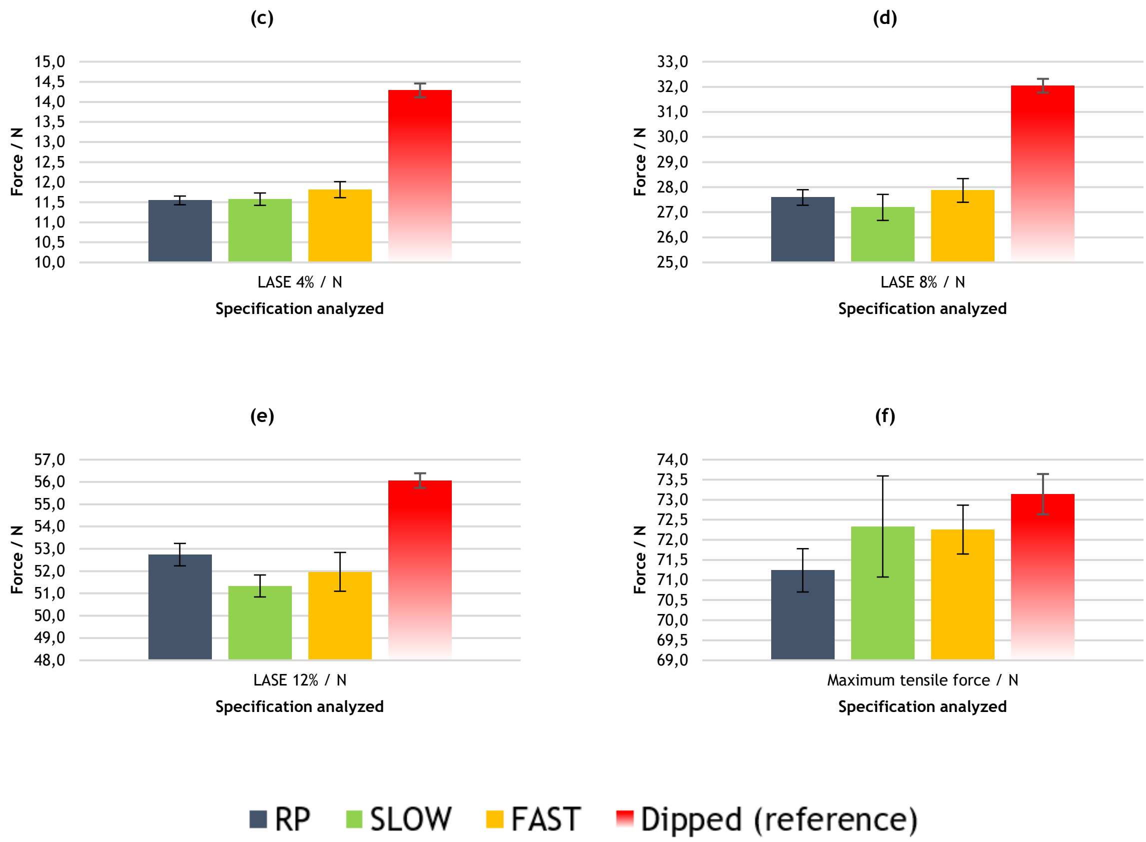
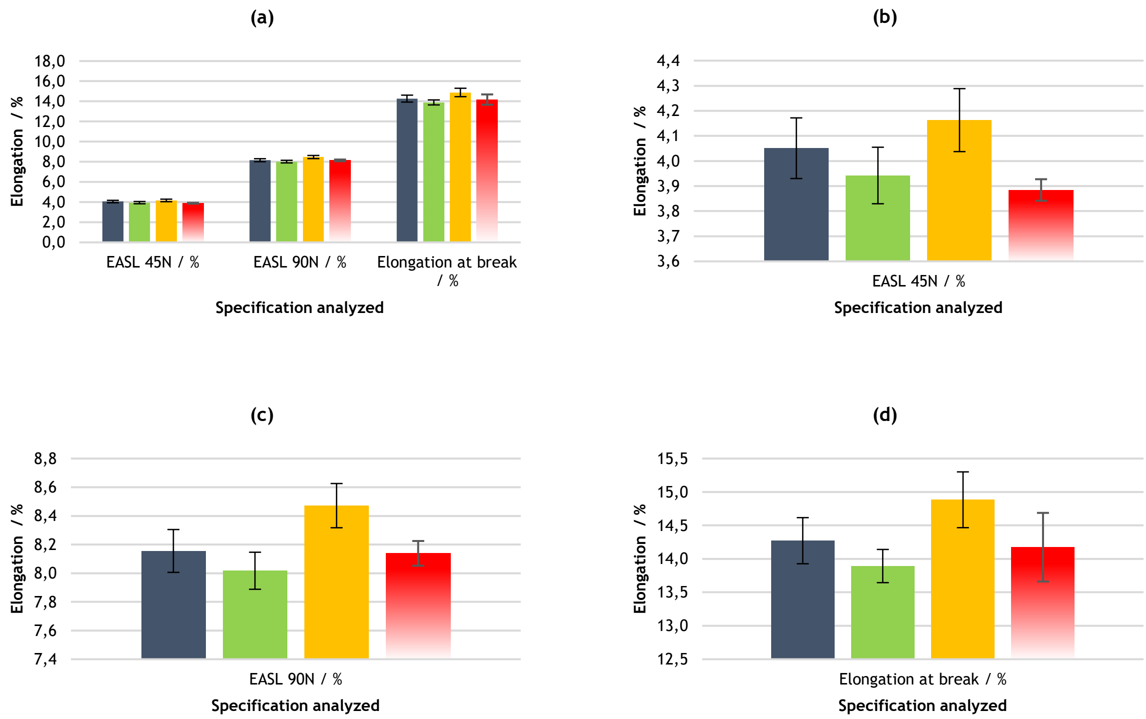


Figure C 10. N1 Force-elongation test results detailed in each specification and their SD.



RP SLOW FAST Dipped (reference)

Figure C 11. P1 Force-elongation test results detailed in each specification and their SD.

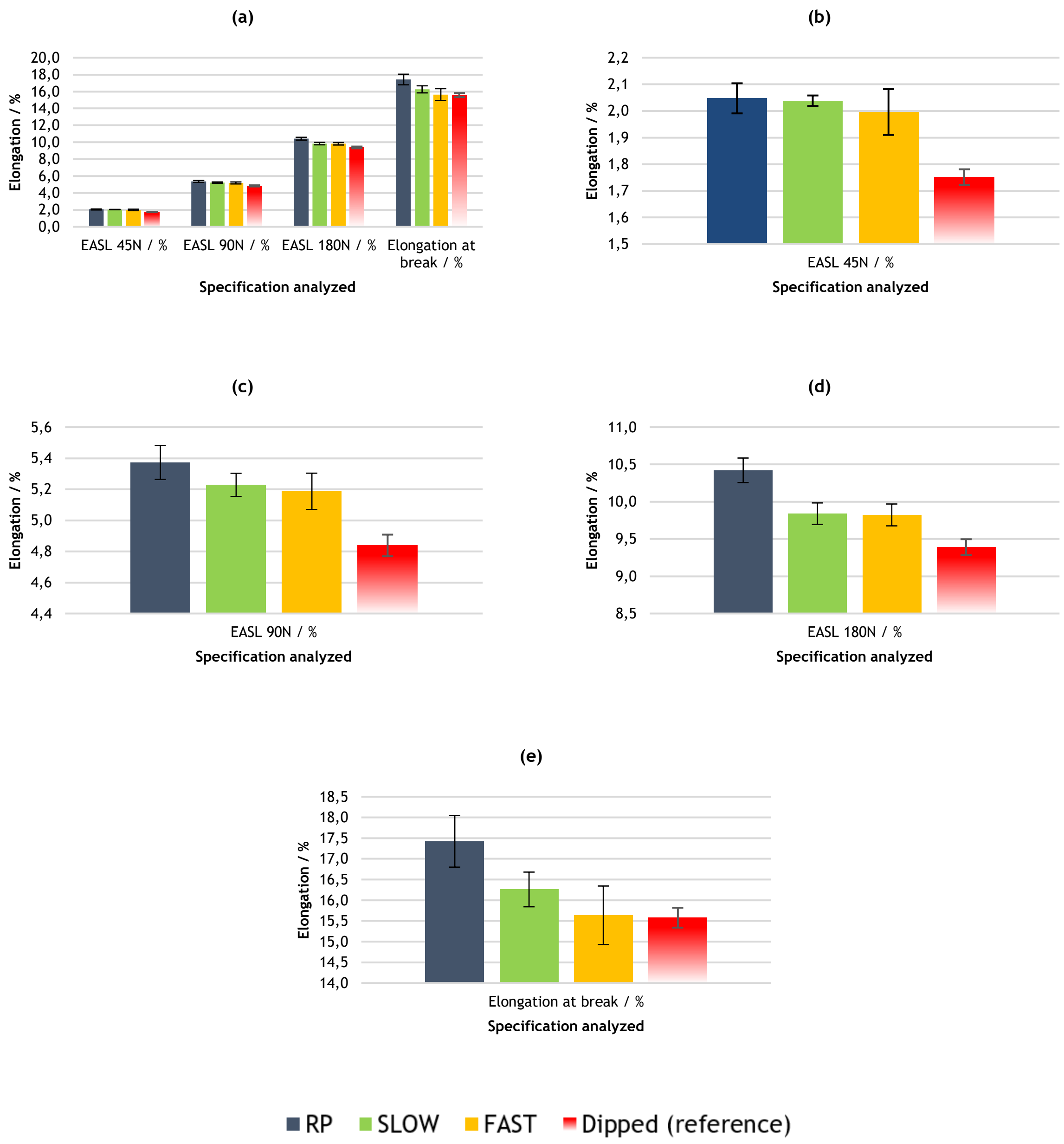


Figure C 12. P2 Force-elongation test results detailed in each specification and their SD.

Method of conditioning

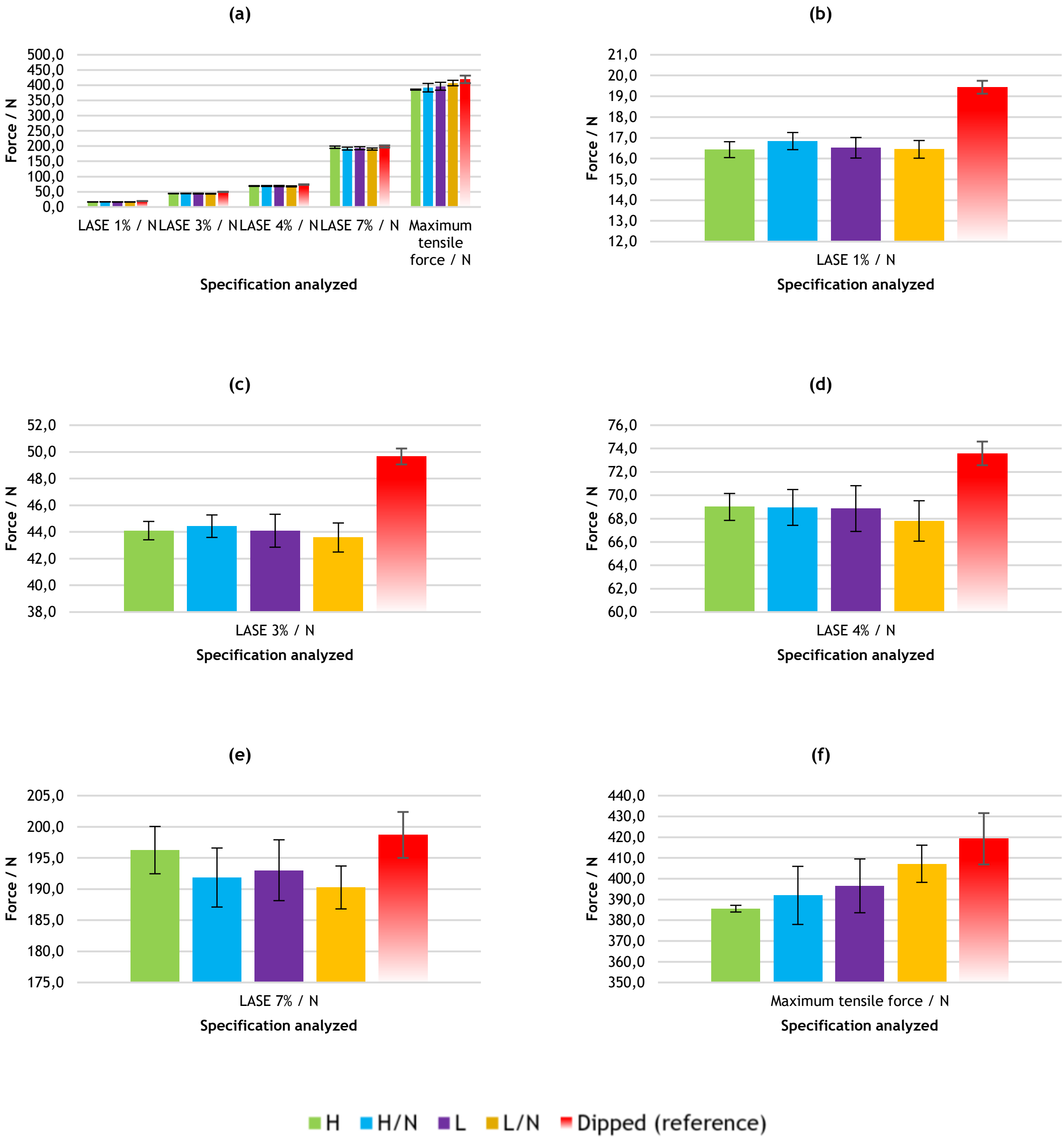
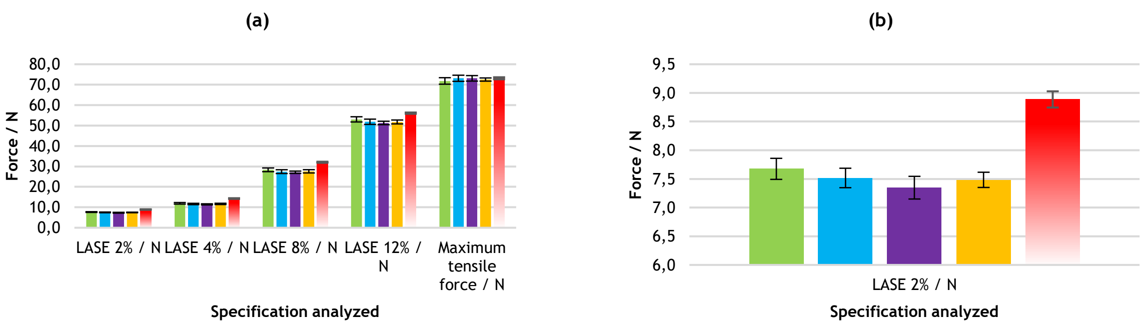


Figure C 13. H1 Force-elongation test results detailed in each specification and their SD.



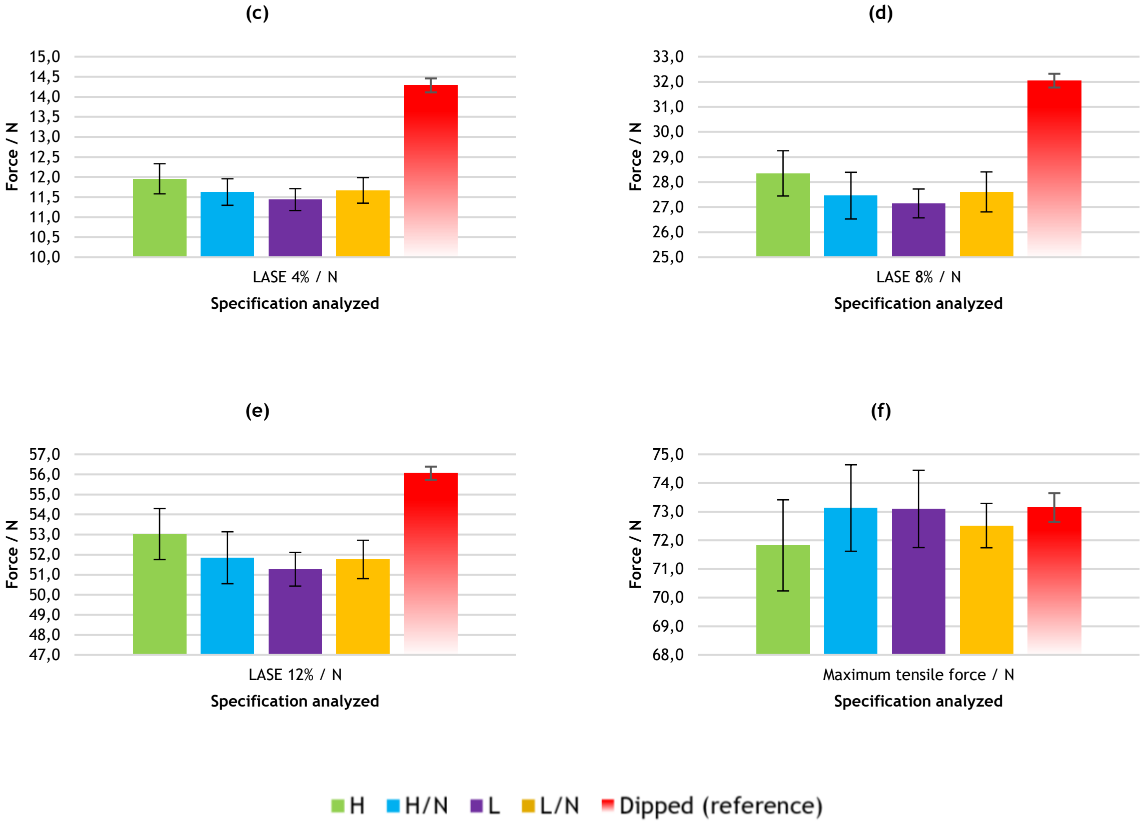


Figure C 14. N1 Force-elongation test results detailed in each specification and their SD.

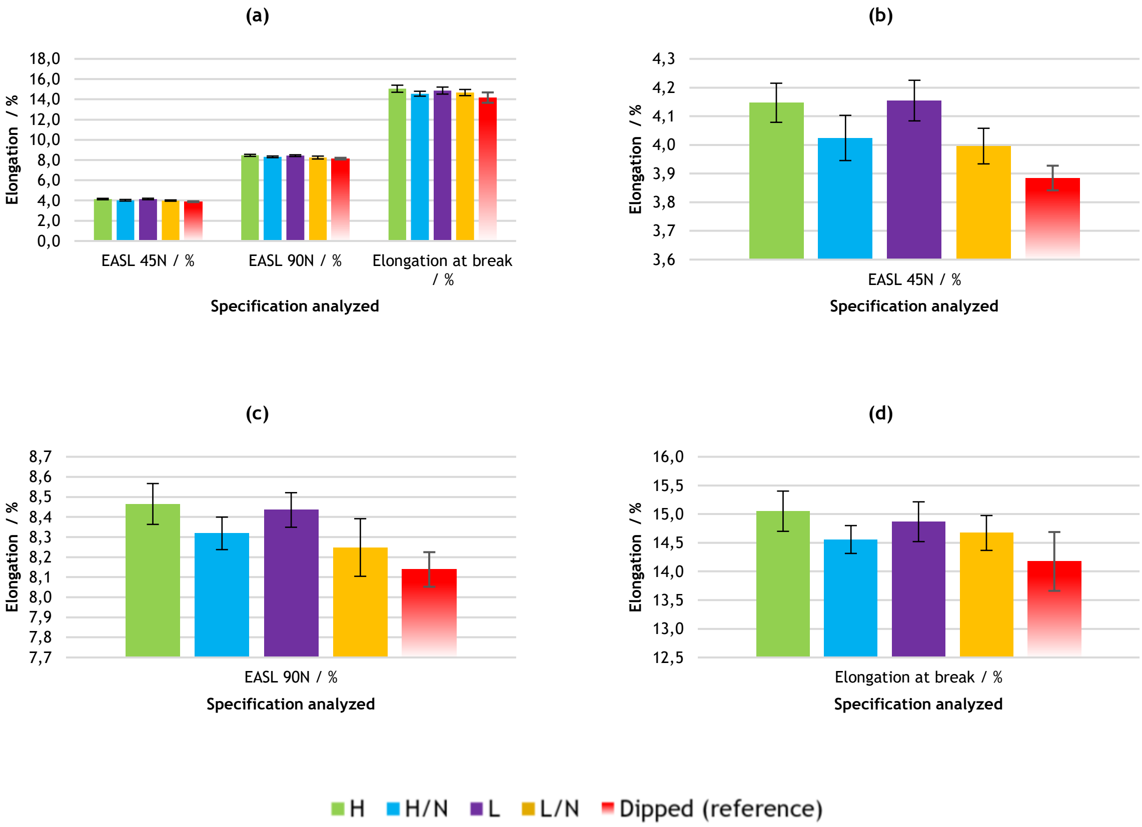


Figure C 15. P1 Force-elongation test results detailed in each specification and their SD.

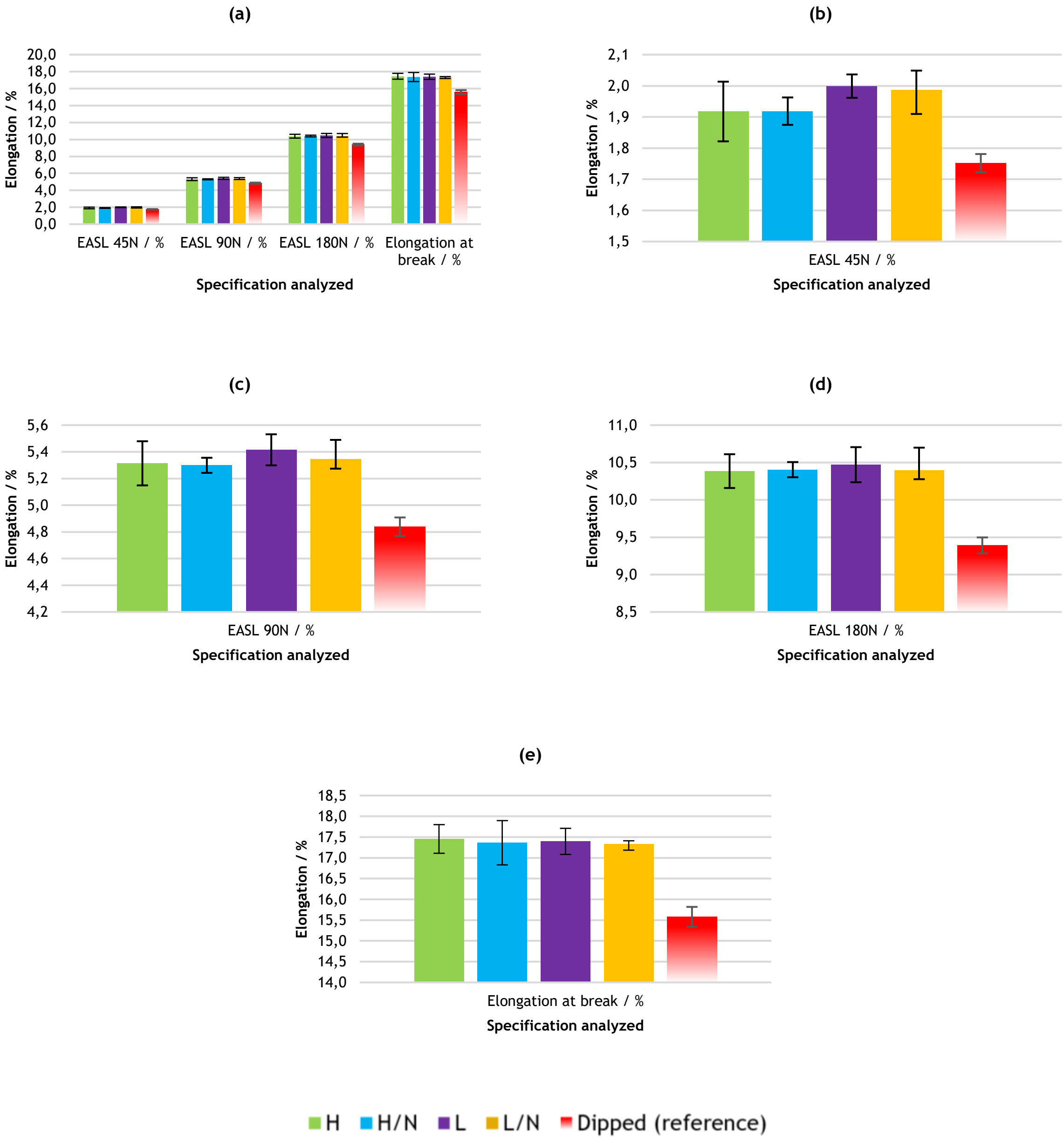


Figure C 16. P2 Force-elongation test results detailed in each specification and their SD.

Appendix D - SWEATY preparation for H1

As mentioned in the development of the study, H1 is a very sensitive material and susceptible to deviations in its values. The material used for the SWEATY preparation is not from the same supplier as for the other preparations, resulting in a different behavior of force-elongation and twist level. Nevertheless, its results will be compared with dipped material and CLEAN preparation from the same supplier in the figure below (Figure D 1**Errore. L'origine riferimento non è stata trovata.**).

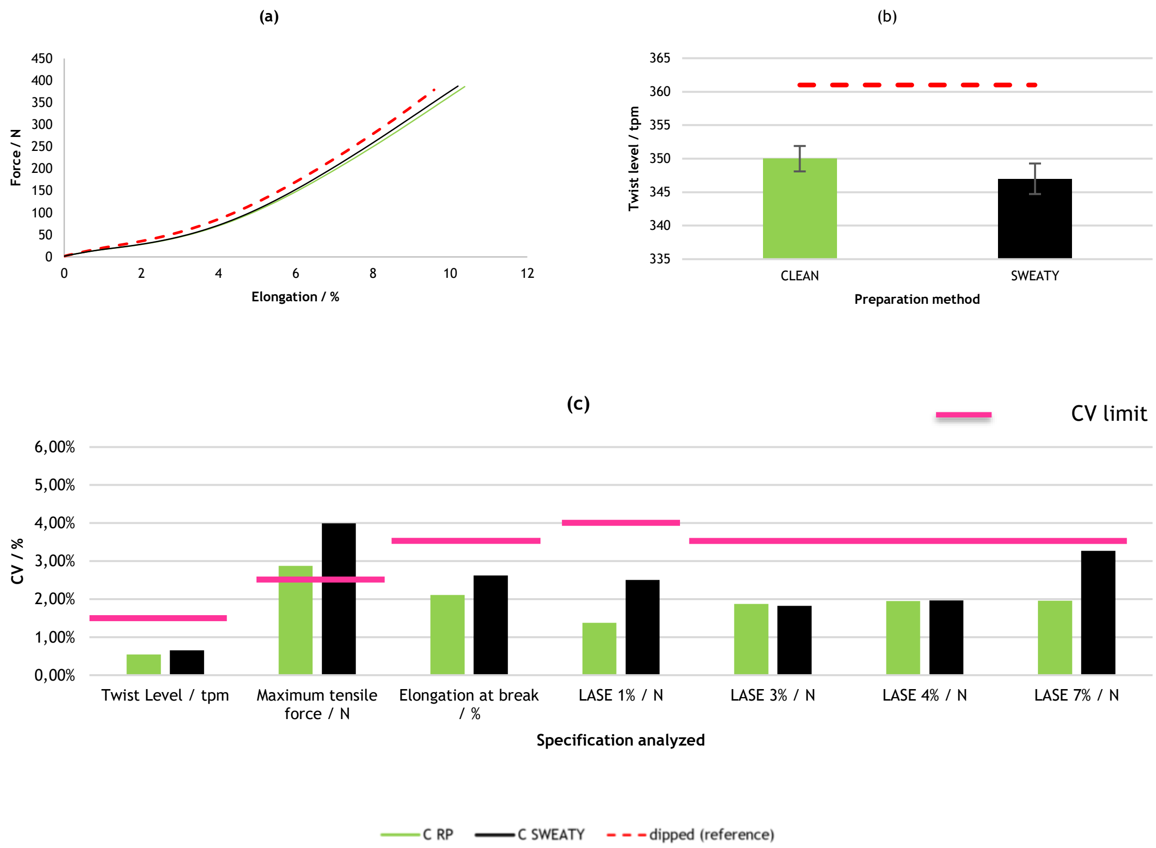


Figure D 1. Test results for H1: (a) force-elongation test, (b) twist level test and (c) CV of manner of preparation test results.

There are no significant deviations in the force-elongation curve (Figure D 1.a), neither in the twist level of the material (Figure D 1.b), which had shown a difference of 3 tpm. With respect to the CV value, all value remained under the established limits for all the specifications, except for maximum tensile force, in which both preparations are above the limits, with SWEATY having the greatest CV value. It also can be observed that there is a tendency for higher deviation for SWEATY when compared to CLEAN.

Appendix E - Comparison of the CV within samples results (direct and conditioned)

This appendix presents a comparison of force-elongation test and twist level test mean deviation before (direct testing – right after pulling the cord out of the rubber) and after conditioning (conditioned) for the materials tested in the first part of the study (Figure E 1, Figure E 2, Figure E 3 and Figure E 4). It can be observed that for the cap strip materials there is a strong tendency of lower force-elongation CV values (Figure E 1.a and Figure E 2.a) and of slightly higher twist level CV values (Figure E 1.b and Figure E 2.b) for conditioned testing, certainly with some exceptions. The higher deviations of conditioning for twist level fit with the facts once the cord is more relaxed, and prone to relax the twist force as well.

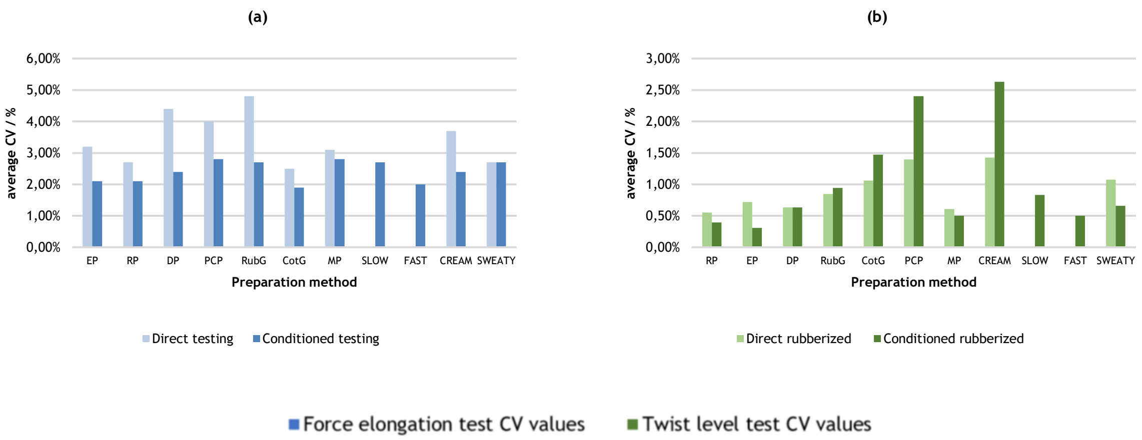


Figure E 1. CV values in force-elongation (a) and twist level (b) tests of H1.

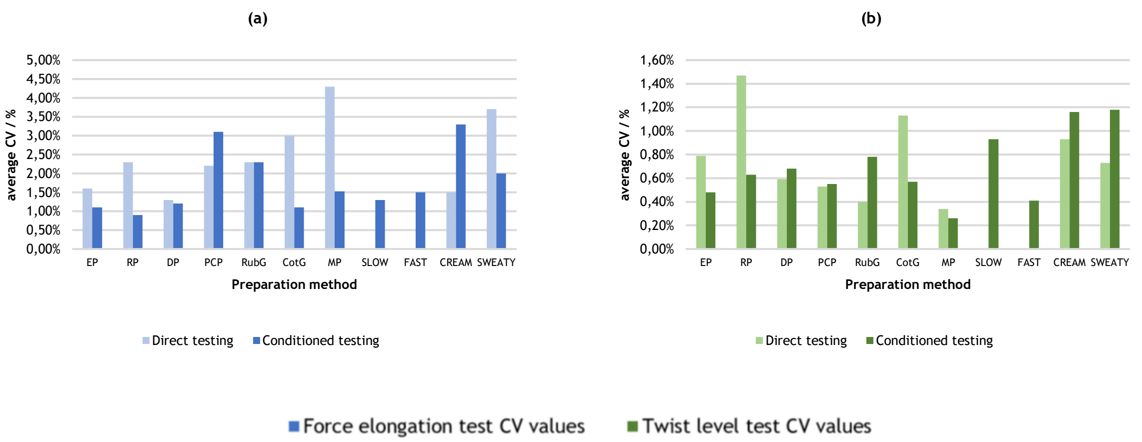


Figure E 2. CV values in force-elongation (a) and twist level (b) tests of N1.

As for the calendered materials, different from the previous materials, the strong tendency of lower CV values for conditioned testing is observed for both force elongation CV values (Figure E 3.a and Figure E 4.a) and twist level CV values (Figure E 3.b and Figure E 4.b). There are, however, some exception that cannot be interrelated since it is different for each material and test.

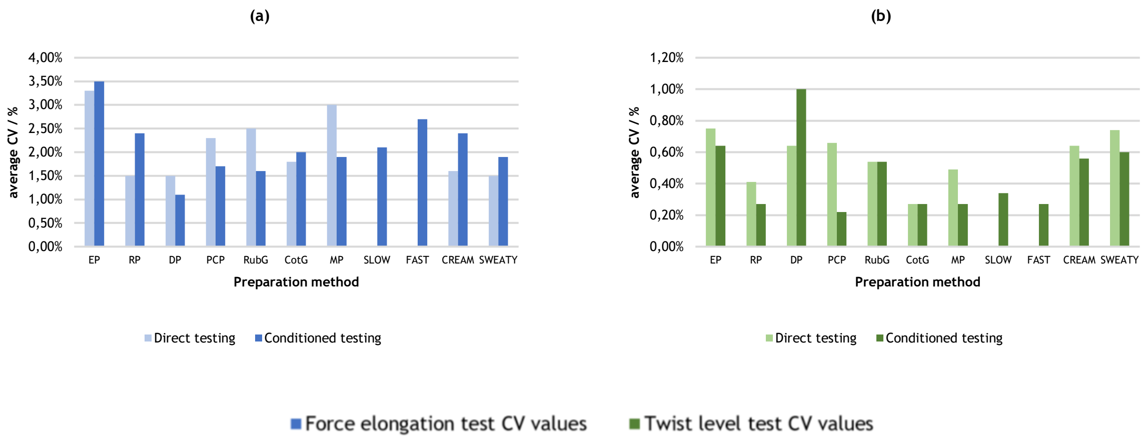


Figure E 3. CV values in force-elongation (a) and twist level (b) tests of P1.

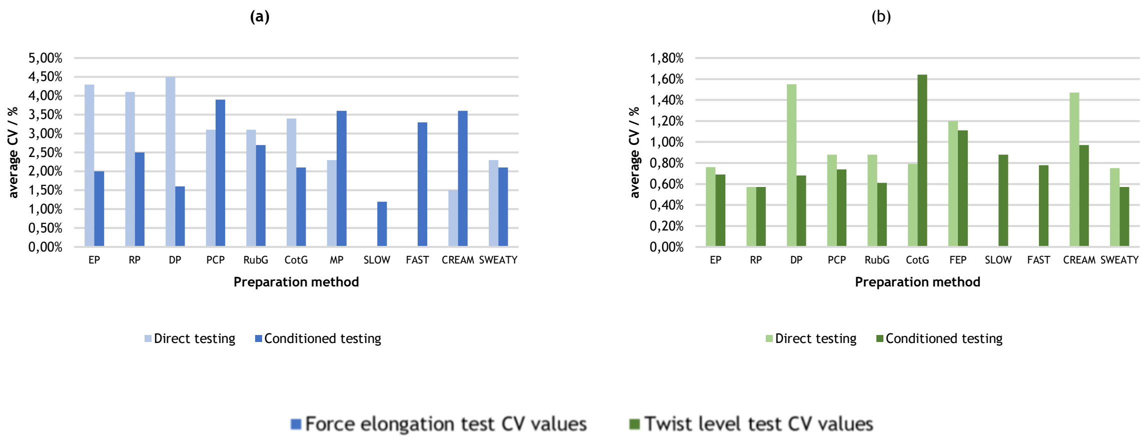


Figure E 4. CV values in force-elongation (a) and twist level (b) tests of P2.

Appendix F - Cross-check results

In this appendix is presented the test results and analysis for dipped materials, as well as the summarized results. Both dipped materials, as expected, exhibited more similar behavior within the different laboratories.

Concerning N2, since it comes in fabric structure, it was only tested after conditioned as the guidelines determines (Figure F 1**Errore. L'origine riferimento non è stata trovata.**). All the laboratories obtained similar and comparable values in force-elongation tests (Figure F 1.a), making it possible the comparison for the results.

The same similarities have been observed for twist level (Figure F 1.b), and as for the rubberized material, there is tendency for lower twist level values for the other laboratories, despite that the difference is not substantial.

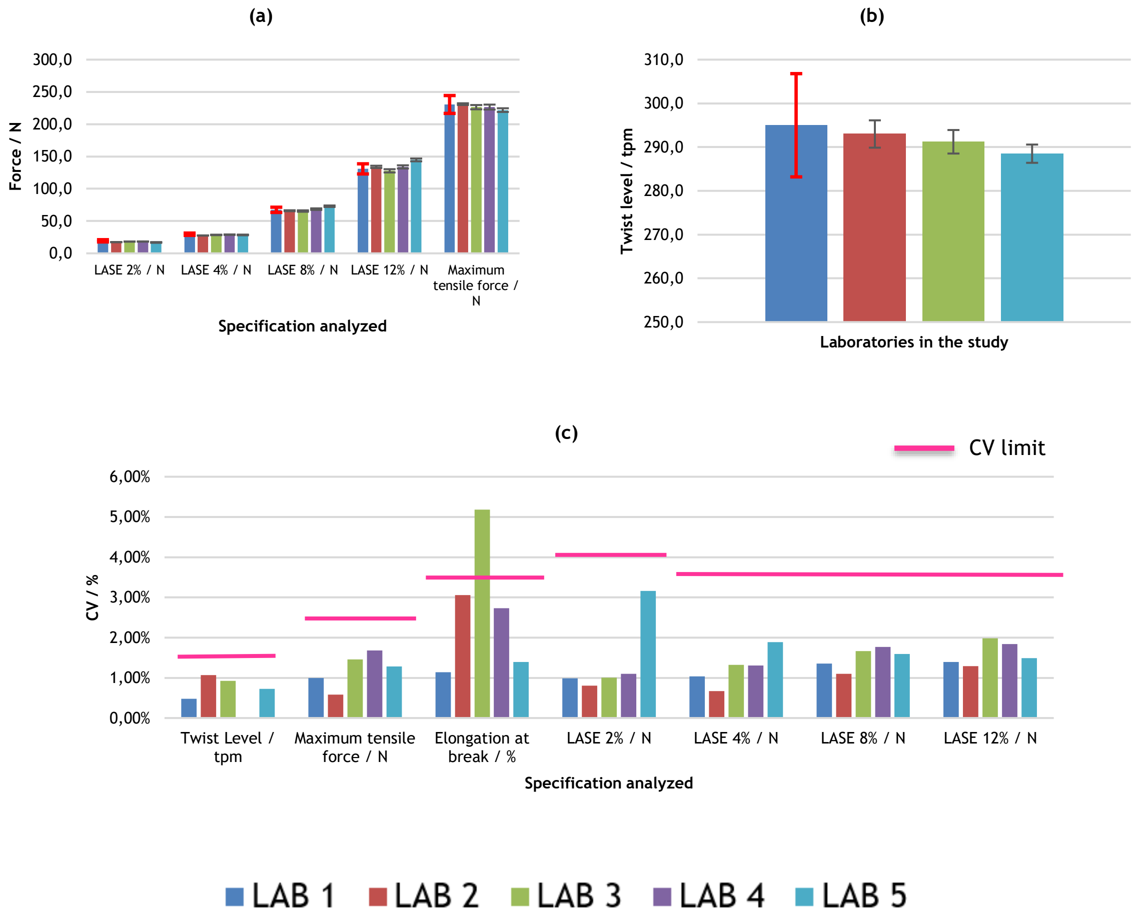


Figure F 1. Force-elongation and twist level test results for N2 dipped, (a) and (b), and their CV values (c) respectively.

As for H1 (Figure F 2**Errore. L'origine riferimento non è stata trovata.**), the material was tested in two different conditioning: the first, the sample were conditioned on the spool and unwounded direct to test, the second, the samples were conditioned on a flat surface. As for the N2, the results were alike and consistent within the laboratories. Despite that, there is still some inconsistency for LASE 7% and Maximum tensile force, for both direct and conditioned samples (Figure F 2.a and Figure F 2.b). Regarding the twist, no significant differences were observed.

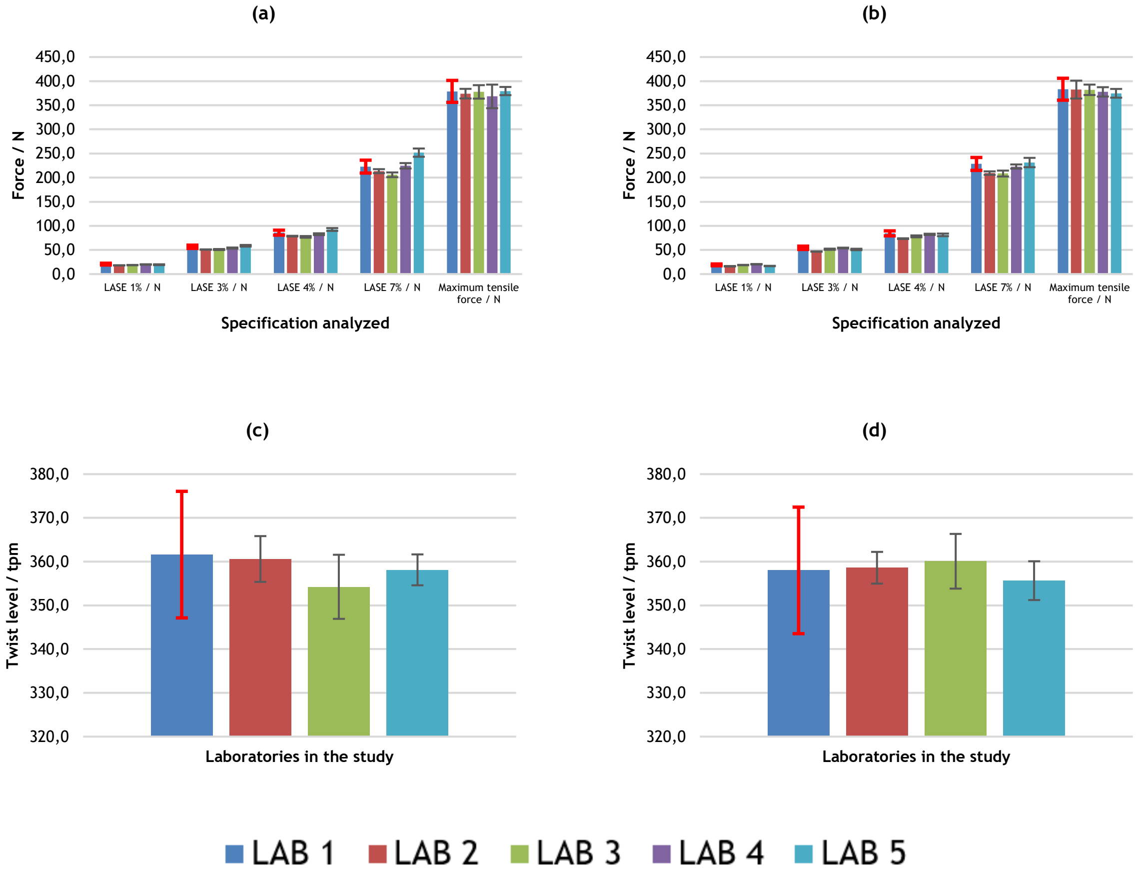


Figure F 2. Force-elongation and twist level test results for H1 dipped, direct (a and c) and conditioned (b and d), respectively.

Regarding the CV for H1 dipped (Figure F 3**Errore. L'origine riferimento non è stata trovata.**), a lower CV value can be observed when compared to the rubberized material, as expected, since the material has not undergone the rubberizing processes and has not suffered any preparation impacts, especially from pulling out the cord from the compound.

In some laboratories, maximum tensile force still has CV values exceeding the limits, as it happened often with rubberized sample.

And once again, laboratory 5 is exhibiting the highest deviations, and some of the above the limits or very close to it. Such a fact, which should be further investigated.

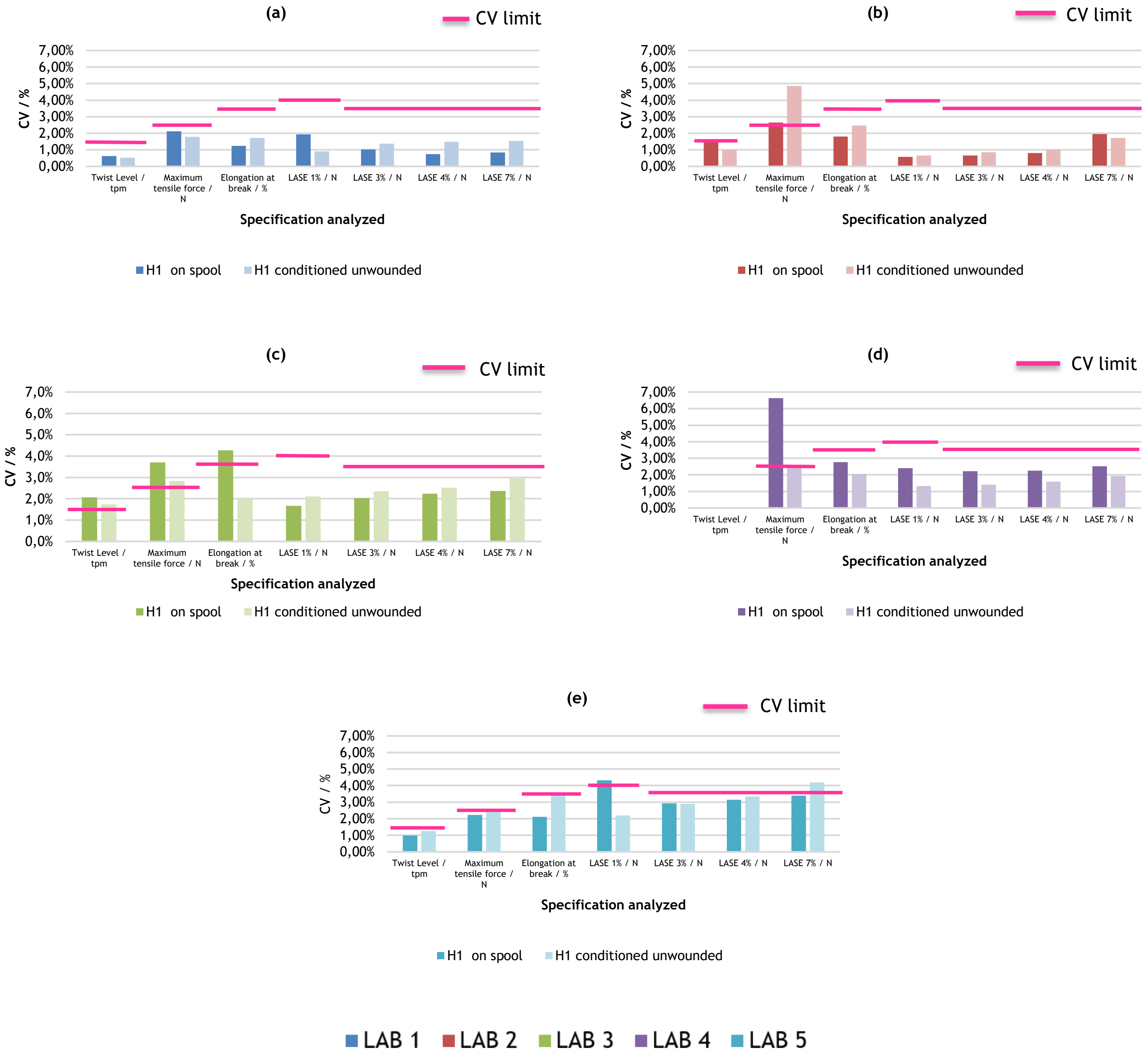


Figure F 3. Force-elongation and twist level CV values for H1 dipped.

It cannot be assumed in this case that there is a tendency for materials conditioned on a flat surface to have lower CV values than the ones conditioned on spool. The behavior is quite different for each laboratory and only laboratory 4 confirms this tendency. Laboratory 3 also displays in part this difference but only for Twist level, maximum tensile force and elongation at break.

When joining the information of the test results and their respective CV values and taking into consideration that the aim was to obtain a comparable result between the laboratories, the results of samples conditioned on a surface seem to be more favorable.

Moving forward, in order to assist the analysis of the tests results, their detailed values are described in the tables below. Respectively, Table F 1 for N2 dipped material, Table F 2**Errore. L'origine riferimento non è stata trovata.** for H1 dipped material, Table F 3 for N2 rubberized material and Table F 4**Errore. L'origine riferimento non è stata trovata.** for H1 rubberized material.

Table F 5 displays the specification limits for the results for N2 material and Table F 6**Errore. L'origine riferimento non è stata trovata.**, for H1 material.

Table F 1. Test results for N2 dipped material.

Test method	LAB 1 (reference lab)	LAB 2	LAB 3	LAB 4	LAB 5
Twist Level / tpm	295.0	293.0	291.2	-	288.5
Twist Level / tpm	301.4	290.6	296.0	-	289.7
Maximum tensile force / N	230.6	231.1	226.4	226.5	221.9
Elongation at break / %	21.3	20.4	20.9	20.7	19.1
LASE 2% / N	19.4	17.3	18.2	18.3	16.9
LASE 4% / N	29.6	27.6	28.5	28.9	28.5
LASE 8% / N	67.4	66.0	65.5	68.4	73.0
LASE 12% / N	130.8	133.9	127.8	133.9	144.7

Table F 2. Test results for H1 dipped material.

Test method	H1 on spool					H1 conditioned unwinded				
	LAB 1 (reference lab)	LAB 2	LAB 3	LAB 4	LAB 5	LAB 1 (reference lab)	LAB 2	LAB 3	LAB 4	LAB 5
Twist Level / tpm	361.6	360.6	354.2		358.1	358.0	358.6	360.1		355.7
Twist Level / tpm	362.4	361.0	352.2		370.5	362.4	360.6	356.0		371.9
Maximum tensile force / N	378.8	374.1	377.7	368.4	379.5	383.2	382.5	382.0	377.7	374.8
Elongation at break / %	9.6	9.9	10.2	9.4	8.8	9.4	9.9	10.0	9.5	9.1
LASE 1% / N	20.6	18.1	18.6	19.9	19.4	19.1	16.2	18.7	20.3	16.7
LASE 3% / N	56.6	50.9	50.9	53.9	58.7	54.5	46.8	51.5	54.0	51.0
LASE 4% / N	85.9	78.7	77.1	82.7	92.4	84.4	73.4	78.2	82.4	81.3
LASE 7% / N	222.8	213.4	205.8	224.4	251.8	228.4	209.4	208.4	223.0	231.3

Table F 3. Test results for N2 rubberized material.

Test method	N2 direct					N2 conditioned				
	LAB 1 (reference lab)	LAB 2	LAB 3	LAB 4	LAB 5	LAB 1 (reference lab)	LAB 2	LAB 3	LAB 4	LAB 5
Twist Level / tpm	296.4	295.6	291.0		288.5	295.0	297.2	288.7		287.1
Twist Level / tpm	303.2	296.4	298.7		291.5	296.2	293.6	295.8		289.6
Maximum tensile force / N	231.6	235.8	229.9	227.2	229.8	227.9	231.9	223.4	224.8	228.9

Elongation at break / %	20.6	20.6	20.3	19.6	20.0	20.4	20.1	19.8	19.4	19.8
LASE 2% / N	19.8	17.6	17.8	19.6	17.8	19.1	16.8	16.6	17.8	17.3
LASE 4% / N	30.2	27.8	28.4	30.4	28.3	29.3	26.7	26.7	28.0	28.0
LASE 8% / N	68.5	65.4	66.1	70.0	67.4	67.8	63.6	63.0	65.7	68.0
LASE 12% / N	133.3	133.1	131.7	136.9	135.2	132.9	131.1	128.4	132.7	137.8

Table F 4. Test results for N2 rubberized material.

Test method	H1 direct					H1 conditioned				
	LAB 1 (reference lab)	LAB 2	LAB 3	LAB 4	LAB 5	LAB 1 (reference lab)	LAB 2	LAB 3	LAB 4	LAB 5
Twist Level / tpm	355.8	354.8	341.5		316.2	350.6	345.2	333.8		331.7
Twist Level / tpm	361.6	362.0	353.1		367.9	361.6	360.4	357.1		370.6
Maximum tensile force / N	394.8	358.6	371.0	362.0	382.0	386.2	354.0	368.6	366.8	368.4
Elongation at break / %	10.4	9.6	10.0	9.1	8.9	10.4	9.7	10.9	9.5	8.7
LASE 1% / N	19.9	18.6	12.0	18.2	18.4	16.9	13.6	10.9	14.2	15.6
LASE 3% / N	50.6	47.6	37.4	49.1	53.1	45.3	38.2	34.0	40.2	46.5
LASE 4% / N	77.5	73.7	58.7	77.6	84.8	70.6	61.4	53.4	64.6	76.7
LASE 7% / N	209.2	208.2	184.7	228.7	247.6	197.3	189.8	174.2	204.7	241.7

Table F 5. Specification limits for N2.

	Specification range (±)	
Specification	N2 dipped	N2 rubberized
Twist Level / tpm	17.7	17.8
Maximum tensile force / N	21	21
Elongation at break / %	1.9	1.9
LASE 2% / N	2.6	2.7
LASE 4% / N	2.7	2.7
LASE 8% / N	6.1	6.2
LASE 12% / N	11.8	12.0

Table F 6. Specification limits for H1.

	Specification range (±)	
Specification	H1 dipped	H1 rubberized
Twist Level / tpm	21.7	21.3
Maximum tensile force / N	34	36
Elongation at break / %	0.9	0.9
LASE 2% / N	2.8	2.7
LASE 4% / N	5.1	4.6
LASE 8% / N	7.7	7.0
LASE 12% / N	20.1	18.8