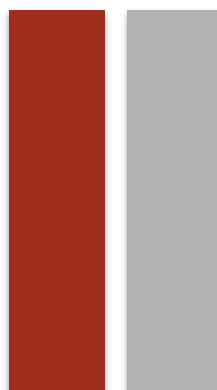


Design of Bio Based Coated Textiles with Incorporation of Pine Wood Wastes Experimental Leather Alternatives

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Advisor: Prof. Doutor Jorge Lino

In coordination with: CITEVE

CITEVE advisor: Augusta Silva

To my Dearing parents

Abstract

This dissertation is the result of a collaborative effort between CITEVE (Technological Center for the Textile and Clothing Industries) and the Faculty of Engineering at the University of Porto (FEUP). It constitutes a part of the larger international project entitled "Diseño de una nueva arquitectura de materiales activos, vivos y adaptativos, basados en principios de la biomimesis y la revalorización de residuos industriales del sector manufacturera de madera de Chile" initiated by Universidad del Bío-Bío in Chile.

The primary objective of this study was to develop sustainable coated materials using pine wood sawdust waste sourced from Chile and pine bark waste obtained from Portugal. These wastes were incorporated in the creation of experimental coated fabrics, employing biomimetic principles that hold the potential to serve as viable alternatives to traditional leather. Additionally, this research aimed to establish a value chain for the wood waste, thus contributing to the initiation of a circular economy paradigm. Furthermore, another objective was the creation of a showcasing object that exemplifies the diverse applications of the developed coated materials.

This document main content is divided in four chapters: the Theoretical framework where all the investigation and research that support this project are presented; Material development, elucidates all the procedures and experiments used and done to reach the final coated materials; Material and formulations testing that details the tests conducted on the materials and their respectful formulations as well as presents the corresponding results and finally the Product development that delineates the creative and production processes involved in the design of an object utilising the developed coated materials.

Based on the findings, it can be concluded that, while further enhancements and testing are still required, the creation of experimental coated materials from the aforementioned wood wastes, as proposed in this study, was achieved and represents a promising and viable alternative to conventional leather.

Keywords: Design; Material Design; Coated Textiles; Leather Alternatives; Wood Waste

Resumo

Esta dissertação é resultado da colaboração entre o CITEVE (Centro Tecnológico das Indústrias Têxteis e do Vestuário) e a Faculdade de Engenharia da Universidade do Porto (FEUP). Constitui parte de um projeto maior internacional intitulado "Diseño de una nueva arquitectura de materiales activos, vivos y adaptativos, basados en principios de la biomimesis y la revalorización de residuos industriales del sector manufacturera de madera de Chile" iniciado pela Universidad del Bío- Bío no Chile.

O objetivo principal deste estudo foi o desenvolvimento de materiais têxteis revestidos mais sustentáveis através da utilizando de resíduos de serrim de madeira de pinho provenientes do Chile e resíduos de casca de pinho obtidos em Portugal. Estes resíduos foram incorporados na criação de materiais têxteis revestidos experimentais, empregando princípios de biomimética tornando-os potenciais alternativas ao couro tradicional. Adicionalmente, esta investigação teve como objetivo estabelecer uma cadeia de valor para os resíduos de madeira, contribuindo assim para a iniciação de um paradigma de economia circular. Além disso, outro objetivo foi a criação de um produto que exemplificasse as diversas aplicações dos materiais revestidos desenvolvidos.

O conteúdo principal deste documento está dividido em quatro capítulos: o Enquadramento teórico onde são apresentadas todas as investigações e pesquisas que suportam este projeto; o Desenvolvimento dos materiais revestidos, que elucida todos os procedimentos utilizados e experiências realizadas para chegar aos materiais revestidos finais; Ensaios de materiais e das formulações que detalham os testes realizados nos materiais e respetivas formulações bem como apresenta os resultados destes testes, e por fim, o Desenvolvimento do produto que apresenta os processos criativos e de produção envolvidos na conceção do produto desenhado utilizando os materiais revestidos desenvolvidos.

Com base nos resultados obtidos, pode concluir-se que, embora sejam necessárias melhorias e mais ensaios, foi possível desenvolver-se os materiais têxteis revestidos experimentais utilizando os resíduos de madeira mencionados acima, conforme foi proposto, representando uma alternativa promissora e viável ao couro convencional.

Palavras-chave: Design, Design de materiais; Têxteis revestidos; Alternativas ao couro; Resíduos de madeira

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“I always get to where I’m going by walking away from where I have been.”

- A.A. Milne

Table of Contents

Abstract.....	9
Resumo	10
Acknowledgments.....	12
Table of Contents.....	15
List of Figures	18
List of Tables	21
Abbreviations and Symbols.....	22
1. INTRODUCTION	24
1.1. Context.....	25
1.2. Objectives.....	25
1.3. Adopted structure and methods	26
1.3.1. Methodology for the Theoretical Framework	26
1.3.2. Methodology for the New Coated Material Development.....	26
1.3.3. Methods for the designed materials formulations and surface tests	27
1.3.4. Methods to the Showcasing Object Development	28
2. THEORETICAL FRAMEWORK	30
2.1. Introduction	31
2.2. Design and Sustainability	32
2.2.1. Circular Design and Circular Economy	32
2.3. The world's use of resources and waste generation and management.....	36
2.4. Wood Waste in Chile and Portugal	38
2.5. The textile coating technique	39
2.5.1. The Coating Methods.....	40
2.6. Polymers and Biopolymers used in the coating industry.....	43
2.7. Biomimetics.....	45
2.8. Active, living, and adaptive materials	45
2.9. The leather industry.....	46
2.10. Alternatives to Leather	47
2.10.1. Synthetic Leather	47
2.10.2. Bio- Based Leather Alternatives.....	47
2.10.3. Lab-cultured leather.....	51
2.11. Chapter's summary	52
3. DESIGN OF THE COATED FABRICS USING PINE WOOD WASTES.....	54
3.1. Introduction	55

3.2.	Previous steps to coating	57
3.2.1.	Drying the pine bark.....	57
3.2.2.	Grinding and sieving the pine bark	57
3.2.3.	Grinding the pine sawdust	58
3.2.4.	Sift the pine bark and sawdust powders.....	59
3.2.5.	Ball milling the pine bark	59
3.3.	Coating formulation and application	60
3.3.1.	Formulations	60
3.3.2.	Weight measurements.....	61
3.3.3.	Mixing components	61
3.3.4.	Measurement of viscosity (and pH).....	61
3.3.5.	Strain the formulation.....	63
3.3.6.	Fabric fastening and knife adjustments.....	63
3.3.7.	Textile Coating Operation	64
3.3.8.	Drying.....	64
3.3.9.	Topcoat	65
3.3.10.	Curing	65
3.3.11.	Calendering and Embossing	65
3.4.	New Coating Materials Development.....	66
3.5.	Conclusions on the material development.....	75
4.	DESIGNED MATERIALS FORMULATIONS AND SURFACE TESTS.....	77
4.1.	Introduction	78
4.2.	Thermogravimetric Analysis (TGA)	78
4.2.1.	TGA Results	79
4.3.	Differential Scanning Calorimetry (DSC)	80
4.3.1.	DSC Results.....	80
4.4.	Fourier Transform Infrared Spectroscopy (FTIR)	81
4.4.1.	FTIR Results	82
4.5.	Colour fastness to washing and colour fastness to direct sunlight exposure.....	82
4.5.1.	Colour analysis of the sunlight exposure and washing cycles tests.....	84
4.5.2.	Results and conclusions of the sunlight exposure tests	86
4.5.3.	Results and conclusions of the washing exposure tests.....	89
4.6.	Martindale abrasion resistance tests (ISO 5470-2).....	92
4.6.1.	Results of the Martindale abrasion test.....	92
4.7.	Colour fastness to rubbing test (ISO 105-X12).....	94
4.7.1.	Results of the colour fastness to rubbing test	95

4.8.	Colour fastness to artificial light: Xenon arc fading lamp test (ISO 105-B02:2014).....	96
4.8.1.	Results of the colour fastness to artificial light test.....	97
4.9.	Sewability testing.....	98
4.9.1.	Results of the sewability test.....	99
5.	SHOWCASING OBJECT DEVELOPMENT.....	101
5.1.	Object's inspiration.....	102
5.1.1.	Moodboard.....	102
5.1.2.	Designing.....	104
5.2.	The petals.....	104
5.3.	The base and shape of the purse.....	107
5.4.	The sewing patterns and final mockup.....	109
5.5.	The prototype.....	111
5.5.1.	Making of the prototype.....	111
6.	CONCLUSIONS AND FUTURE WORK.....	121
6.1.	Conclusions.....	122
6.2.	Final considerations, limitations and future work.....	122
7.	REFERENCES.....	124
8.	APPENDIX.....	127

List of Figures

Figure 1: Schematic diagram of Circular design. (adapted from The Circular Design Guide, n.d.).....	34
Figure 2: Schematic diagram of the relationship and differences between circular economy and ecodesign. (image from Prendeville, S. & Sherry, J., 2014)	34
Figure 3: Schematic diagram of the Industrial Ecology concept. (image from Celik B.,2013)	35
Figure 4: Upcycling, Recycling, and Downcycling symbols. (image from Gayla, n.d.)	36
Figure 5: (1) Example of products used in a coating formulation and (2) an image of how the paste using these products looks like. (images from the author)	41
Figure 6: Schematic diagram of the knife-over-roll coating application. (1) knife; (2) roll; (3) substrate; (4) coating material. (adapted from Sen, A., 2008)	42
Figure 7: Example of an embossing. (1) The pattern used to create the texture on the surface; (2) the coated surface after the embossing process. It is visible where it was applied and where it wasn't. (images from the author).....	43
Figure 8: Schematic explanation of the difference between synthetic polymers; bio-based polymers and biopolymers with examples of each. (adapted from Plastics NZ, 2023)	44
Figure 9: Leonardo Da Vinci's drawings on flying mechanisms. (images from Da Vinci, L., 1488).....	45
Figure 10: Example Leather alternative made from pineapple leaves. (image from Ananas Anam, n.d.)	45
Figure 11: Hugo Boss Shoes line made with Piñatex®. (image from Hugo Boss, 2018).....	49
Figure 12: Fruitleather x Windmilkey collaboration. (image from Fruitleather Rotterdam, n.d.).....	49
Figure 13: Mylo™ x Adidas Stan Smith made with mycelium-based leather alternative. (image from Mylo, 2021).....	50
Figure 14: Karl Lagerfeld X Amber Valletta handbag made with Desserto® leather alternative. (image from Karl Legarfeld, 2021)	51
Figure 15: Black Gala Backpack from Allégorie made with AppleSkin™ leather alternative. (image from Allégorie, n.d.).....	51
Figure 16: Modern Meadow's Lab- Cultured Leather. (image from Lampoon, 2021).....	51
Figure 17: VitroLabs Inc. Lab- Cultured Leather. (image from Lampoon, 2021)	52
Figure 18: Schematic diagram of the steps of the Material Development. (created by the author with miro tool)	56
Figure 19: Drying and curing tunnel. (image from Acosgraf, n.d.).....	57
Figure 20: Retsch SM 300 Cutting mill machine. (image from Retsch, n.d.).....	57
Figure 21: Bottom sieves for the cutting mill; (1) Sieve with square holes with 10 mm; (2) Sieve with square holes with 4 mm; (3) Sieve with trapezoid holes with 1 mm; (4) Sieve with trapezoid holes with 0.25 mm. (images from the author)	57
Figure 22: Pine bark throughout the different stages of the cut milling process; (1) initial state; (2) after milling with the sieve with 10 mm holes; (3) after milling with the sieve with 4 mm holes; (4) after milling with the sieve with 1 mm holes; (5) after milling with the sieve with 0.25 mm holes. (images from the author).....	58
Figure 23: Pine sawdust throughout the different stages of the cut milling process; (1) initial state; (2) after milling with the sieve with 1 mm holes; (3) after milling with the sieve with 0.25 mm holes. (images from the author).....	58
Figure 24: Different views of the Manual sieves used to achieve the 0.2mm particle size. (images from the author)	59
Figure 25: Retsch PM 100 Ball milling machine. (image from Retsch, n.d.)	59
Figure 26: (1) Pine bark and (2) sawdust particle size after the manual sift process. (images from the author)	59

Figure 27: Pine bark particle size after the ball milling process. (image from the author)	60
Figure 28: Precision scale used for measuring the formulations components. (image from LabMakelaar, n.d.)	61
Figure 29: Techmatic S2 Mechanical Agitator. (image from the author)	61
Figure 30: Brookfield DVE Viscometer. (image from the author).....	61
Figure 31: Viscometer display with the indications of its values and function buttons. (image from the author)	62
Figure 32: Spindle set available for the viscometer used. (image from the author).....	62
Figure 33: Example of spindle points with the indication of the indentation. (image from the author)	62
Figure 34: pH equipment, WTW Inolab pH7110 pH Meter. (image from Indiamart, n.d.)	63
Figure 35: Step of straining a formulation. (image from the author).....	63
Figure 36: Mathis AG LABCOATER type «LTE-S», coating application, drying and curing machine. (image from Mathis AG, n.d.)	63
Figure 37: Fabric and knife prepared for coating, indicating the regulators of the knife's height. (image from the author)	63
Figure 38: Display that shows the drying settings of one of the coating applications, and explanation of the values that appear on the display. (image from the author)	64
Figure 39: Application of a coating formulation. (1) Formulation poured on the substrate; (2) spreading. (image from the author)	64
Figure 40: Display that shows the curing settings of a coating applications. (image from the author)	65
Figure 41: Machine's model used to make the calendering and embossing processes. (image from Macpi, n.d.)	65
Figure 42: Images of all the results obtained in the coating experiments. (images from the author). 70	
Figure 43: Sample #PT12. (images from the author).....	72
Figure 44: Sample #PT14.2. (images from the author)	74
Figure 45: Graphics with the results of the TGA tests.(image from FEUP,2023).....	79
Figure 46: Graphics with the results of the TGA tests. Indication of the temperature at which there is a 50% mass loss in each formulation. (image from FEUP,2023).....	80
Figure 47: Graphics with the results of the DSC tests. (image from FEUP,2023)	81
Figure 48: Graphic with the results of the FTIR tests (adapted). (image from FEUP,2023).....	82
Figure 49: Samples that were exposed to direct sunlight. (1) Sample with pine bark waste; (2) Sample with pine sawdust waste. (images from the author).....	83
Figure 50: Machine and product used in the washing exposure tests. (1) washing machine model used; (2) Washing detergent used. (Samsung, n.d. ; Persil, n.d.)	83
Figure 51: (1) Samples of the material designed with pine bark waste that were tested for colour fastness to washing; (2) Samples of the material designed with pine sawdust waste that were tested for colour fastness to washing. (images from the author)	83
Figure 52: Datacolor® Spectraflash® SF450® - the machine that “reads” the samples.	84
Figure 53: Some of the tools and settings used to calibrate Datacolor® Spectraflash® SF450®. (1) Aperture used for the calibration and tests. (2) Settings for the calibration. (images from the author)	84
Figure 54: Patterns to calibrate the machine Datacolor® Spectraflash® SF450®. (1) and (2) different views of the black standard for the calibration; (3) and (4) different views of the white standard for the calibration. (images from the author)	84
Figure 55: Positions in which the samples were read. (images from the author).....	86
Figure 56: Schematic diagram of the Martindale Tester. (adapted from Jerkovic, I., et al., 2010).....	92

Figure 57: Schematic diagram of a crockmeter. (adapted from Textile Pad, 2022)	95
Figure 58: Sewing machine used. (image from the author)	98
Figure 59: Samples with the testing sewings. (images from the author)	98
Figure 60: Variety of pinecones. (image from Gardening Knowhow, n.d.)	102
Figure 61: (1) Pinecone in closed state and open state;(2) Close up on the petals of a pinecone. (image from ArtTower, 2014)	102
Figure 62: Moodboard for the development of the handbag.	103
Figure 63: Initial sketches for the project. (images from the author)	104
Figure 64: Sketches of the “petals” shapes (not in scale nor real size). (images from the author)....	105
Figure 65: First petals mockups. (1) petal number 7; (2) petal number 12; (3) petal number 16. (images from the author).....	105
Figure 66: Sketches of the petals number 7, 12 and 16 resized to +20% and + 50% (Not in scale or real size). (images from the author)	106
Figure 67: Mockups of the petals 7, 12 and 16, respectfully, resized to +20%.(images from the author)	106
Figure 68: Mockup of the chosen shape in the designed material. (images from the author)	107
Figure 69: Sketches of the purse formats and the petals arrangement. (images from the author) ..	107
Figure 70: Final sketch of the purse. (image from the author).....	107
Figure 71: Mockup of the petals if glued to the fabric. (1) front view; (2) side view. (images from the author)	108
Figure 72: Experimental models of the petals if sewed to the fabric. (1) front view; (2) side view. (images from the author).....	108
Figure 73: Mockup of the organisation of the petals in the base. (image from the author).....	108
Figure 74: Sewing pattern designed for the pieces that compose the base of the purse (not in scale nor real size). (image from the author)	109
Figure 75: Sewing pattern designed for the pieces that compose the upper part of the purse (not in scale nor real size). (image from the author).....	109
Figure 76: Sewing pattern designed for the pieces that compose the “petals” (not in scale nor real size). (image from the author)	110
Figure 77: Final mockup of the handbag. (image from the author)	110
Figure 78: Sewing pattern for the base of the purse updated (not in scale nor real size). (image from the author)	111
Figure 79: Macpi 923.37.1610 laser machine. (image from Macpi, n.d.)	112
Figure 80: Laser cutting the petals. (image from the author).....	112
Figure 81: Sewing the petals. (images from the author)	113
Figure 82:Cutting the base. (images from the author)	113
Figure 83:Sewing the bottom petal. (image from the author)	114
Figure 84: Sewing the remaining petals. (image from the author)	114
Figure 85:Cut and sewing the upper part. (images from the author)	115
Figure 86:Making the tube for the cord. (images from the author).....	115
Figure 87:Sew the bases to the upper parts. (images from the author)	116
Figure 88: Making the holes for the cords. (image from the author).....	117
Figure 89: Cutting the tube. (image from the author).....	117
Figure 90: Insert the cords; final prototype. (images from the author)	118

List of Tables

Table 1: Polymeric components that were used in the coating formulations. (table created by the author)	60
Table 2: Other components that were used in the coating formulations. (table created by the author)	61
Table 3: Results of the analysis made in the Datacolor® Spectraflash® SF450® to the designed material with pine bark waste samples that were exposed to sunlight. (table created by the author)	87
Table 4: Results of the analysis made in the Datacolor® Spectraflash® SF450® to the designed material with pine sawdust waste samples that were exposed to sunlight. (table created by the author)	88
Table 5: Results of the analysis made in the Datacolor® Spectraflash® SF450® to the designed material with pine bark waste samples that were exposed to washing cycles. (table created by the author)	90
Table 6: Results of the analysis made in the Datacolor® Spectraflash® SF450® to the designed material with pine sawdust waste samples that were exposed to washing cycles. (table created by the author)	91
Table 7: Results of the Martindale abrasion test on samples of the designed material with pine bark waste (table created by the author)	93
Table 8: Results of the Martindale abrasion test on samples of the designed material with pine sawdust waste. (table created by author)	94
Table 9: Settings of the sewing machine used to perform the sewability test. (table created by the author)	99
Table 10: Materials and tools used to make the prototype. (table created by the author)	112

Abbreviations and Symbols

CO₂ – Carbon Dioxide

cm - Centimeters

cps - Centiplase

g - Grams

kPa – Kilo Pascal;

min.- Minutes

mm - Milimeters

N - Newton

Pa.s – Pascal Second

Rpm – Rotations per minute

% - Percentage

°C – Degrees Celsius

Δ - Delta

1.INTRODUCTION

1.1. Context

This dissertation was developed through a collaboration between CITEVE and FEUP. It is part of an international project called “Diseño de una nueva arquitectura de materiales activos, vivos y adaptativos, basados en principios de la biomimesis y la revalorización de residuos industriales del sector manufacturera de madera de Chile.” This project is originally from Universidad del Bío- Bío in Chile and it has many international institutions associated with the project: FEUP and CITEVE are two of these institutions.

1.2. Objectives

The goals of this international project are to develop new active, live and adaptive materials, by applying biomimetic principles and by incorporating in those new materials, pine wood waste from the wood industry from Chile; and to overall develop materials, through design and engineering, that apply circular economy in their creation, in order to add value to the wood waste and reduce the impact from this industry.

In this dissertation, the main goal was to explore the design of a more sustainable textile coating using the wood waste from Chile, creating a new type of experimental fabric that can become an alternative to leather in the future and add value to the wood waste used to create it. In order to do so, it was necessary to explore some of the existing alternatives.

As this project was developed in Portugal, to enrich the experimental work, pine wood waste from the country was also used to develop an alternative option.

Finally, an additional objective was to create an object that exemplified the diverse applications of the developed materials. This object serves as a tangible representation, showcasing the potential utilisation of this material in various contexts and demonstrating its versatility.

1.3. Adopted structure and methods

This dissertation had four major stages in which it can be divided: investigation, which is all the research done and framework that supported this project; material development; material testing and example product development. Each stage required different methodologies which are described in the following subchapters.

1.3.1. Methodology for the Theoretical Framework

The aim of this dissertation was the development of a new textile material, applying wood waste through a coating technique. This type of application, using waste in coatings, is a quite new method and the achieved solutions are considered as leather alternatives. An exploration and study of related subjects within the scope of this dissertation was important:

- Wood waste management in Chile and Portugal (the countries this dissertation involves);
- Circular economy;
- Coating technique;
- The leather industry and existing alternatives.

Academic research methods were used, researching books and articles on these topics, using the research online engine Google Scholar. When no academic sources could be found for particular topics, an informal search was made to gather information in websites like brand websites, magazines, or general news websites.

1.3.2. Methodology for the New Coated Material Development

After the Theoretical Framework was researched and information was gathered, it was necessary to carry out the coating experiments.

The first step in the development of these leather alternatives solutions was waste adequation, starting by drying the Portuguese pine bark and then milling and sieving it to the desired dimension suitable for the coating application. Observations of the coating process were done to understand the process and to learn how to execute it. After learning the process, initial tests were carried out with different polymers in order to determine those that produced results more consistent with traditional leather physical properties for further testing.

In the next weeks a series of tests were conducted to refine the coating formulation and define: the percentage of waste that should be added; the polymers with higher biobased content that better matched the waste and provided the desired touch; as well as, to determine which additional materials like crosslinker (a fixing agent) and topcoat (a finishing agent) were necessary. In these tests the number and thickness of the coating layers and calendering/embossing options that better fit the desired result were also experimented with.

The development of the alternative to leather using the Chilean Pine Sawdust differed since the process of drying was not necessary and since the experiments with this material were carried out when the formulation of the paste using the pine bark was already established. The formulation starting point for the Chilean Pine Sawdust was based on the already optimised formulation for the Portuguese Pine Bark changing only the waste incorporated.

After this first try, adjustments were made in order to achieve the desired result as the previous development.

This phase concluded with viable formulations for each waste and fabric samples of the applied materials developed.

1.3.3. Methods for the designed materials formulations and surface tests

The next stage of designing these experimental materials was to test their formulations and the materials themselves. The tests carried out to the formulations were TGA, DSC and FTIR while the tests made to the materials were colour fastness to washing, colour fastness to direct sunlight exposure, the resistance to abrasion Martindale surface test (ISO 5470-2), the colour fastness to rubbing test (ISO 105-X12) colour fastness to artificial light exposure (ISO 105-B02:2014) and sewability test.

All the tests were carried out on samples of both experimental developed materials, in order to compare them.

All the detailed description of these tests will be presented in chapter 4.

1.3.4. Methods to the Showcasing Object Development

To conclude an exemplary object using the designed coated materials was developed. The inspiration for the object's concept was the pinecone. This article was sketched, and some mockups were developed using similar appropriate fabric until the final design was stabilised and the prototype was made using the samples of the materials developed.

2. THEORETICAL FRAMEWORK

2.1. Introduction

In this chapter, a theoretical framework of the theme of this dissertation is presented which is sectioned in different subchapters for a better organisation.

In the first section an overview on design and sustainability and how design has an important role on the sustainability of the planet is presented.

The next section presents information on humanity's unruly use of resources and waste generation, and how we should be optimising the use of resources and valuing waste as a resource for production and stresses the importance of the circular economy and circular design in the management of resources and waste.

Subchapter 2.4 focuses on Chilean and Portuguese wood waste which are the base of this project and its experiments, which are, in turn, integrated in an international project from a Chilean university.

The next subchapter presents overall information on the use of textile coating technique, on how this technique works, the different steps and materials used in the project.

Focusing on the coating technique, the section that follows describes polymers and biopolymers and bio-based polymers which are the base for coatings.

Biomimetics is also a relevant issue in this dissertation which is discussed and presented in this chapter.

Right after the biomimetics section the concept of active, living and adaptive materials is presented due to its pertinence to the project.

Since the resulting material in this study is a leather-like fabric, it is important to gather knowledge on the leather industry which is the focus of a section of this chapter, as well as the topic on how traditional leather can be very impactful for the environment, and a section dedicated to the leather alternatives that are already available in the market, discriminating the alternatives that are, or can be, as impactful as synthetic leather from the alternatives that are being used and developed that can be a more sustainable option.

In the end of this chapter there is a summary of all the chapter contents.

2.2. Design and Sustainability

Sustainability is presently one of the most relevant topics in society since the environment's integrity has been in jeopardy for many years. But when did this awareness start? It goes back to the 60's and 70's when people started thinking about ecology and started worrying about the impact of industrial production. What was, until then, a material progress, benefit, and a big advancement for society, started to be seen as the beginning of an environmental crisis (Cardoso, 2008).

Alongside the topic on the preservation of the environment, the discussion on sustainable development, regarding not only environmental consciousness but also humanitarian issues, emerged. From 1980 the United Nations began a series of commissions on these topics, to develop projects that promoted the improvement of the industries, on these matters, involving actions on social equality, healthcare, education, and many other topics that had an impact on a more sustainable development of the industries (Margolin, 2002).

From this point, production sectors as well as societies' environmental and humanitarian consciousness began to change. Industries started implementing more environmentally friendly manufacturing processes as consumers started to demand products less harmful to the environment (Manzini, 2002).

Design plays an important role on this matter. The journey to sustainability begins in the design stage of the product before its production. With this in mind, designers started having a very important role in the development of products that were more responsible and many strategies began emerging to help the development of these new products.

2.2.1. Circular Design and Circular Economy

It is important, before understanding circular design, to understand the concept of Circular Economy on which it is based. There are an immense number of definitions of

Circular Economy proposed by several authors, all of them very similar. We will use here the definition given by the Ellen MacArthur Foundation in 2015, one of the most recognized today:

'A circular economy is one that is restorative and regenerative by design and aims to keep products, components, and materials at their highest utility and value at all times, distinguishing between technical and biological cycles.' (Geisendorf, 2018).

The circular economy is an economic model that aims to produce objects and services in a more sustainable way, considering the consumption and waste of resources. It is based on an optimised management and reduction of the virgin extraction of these resources during the production process, and a reduction of waste at the end of life and disposal of the products.

The European Union has been implementing and introducing this model in Europe since 2015, when it launched a set of ambitious policies to be applied in various sectors, with the aim of “closing the cycle” of products through improvements in reuse and recycling (Alves, 2002) (Jia, Yin, Chen, & Chen, 2020).

To take this model forward, several innovative strategies have emerged to help optimise resources. In this sense, design enters as one of the most important collaborators, alongside the development of new materials since these strategies focus right from the initial moments of the product's life (Alves, 2002). Thus, the concept of circular design arises.

Circular design seeks to replace the current conception of production mindset and behaviour through its different phases: product design, communication design, service design, ... It aims to replace linear models and “end of life” concepts by models in which the entire life of the product is designed right at the beginning, from the material it is produced in, the type and consumption of energy in its production, the waste it generates, the ease with which it is repaired, the way it is transported, ... Thus, renewable energy models are applied, resources that do not use chemicals harmful to health and the environment in their production and processes that seek to reduce the waste of materials, ... (Moreno, De los Rios, Rowe, & Charnley).

In circular design, the business model for which the product is

being developed must be considered, in order to apply a circular design strategy that is appropriate. For example, there are strategies that seek to maintain the economic value of the product during its life cycle, while others adopt resources that can be reintegrated into nature or used by other industries (Moreno, De los Rios, Rowe, & Charnley). Figure 1 presents a schematic diagram of what circular design is.

The application of concepts and strategies for a circular design are an urgent response to an environmental problem of enormous scale because, although there have been efforts since the 60's to encourage industries to evolve into a more responsible production, the volume of global consumption spiked over the last years and the efforts to change the industries could not compensate the increase of the industries environmental impact (Agency, 2015).

The following paragraphs describe examples of strategies based on circular economy and design.

Ecodesign

Ecodesign is a project development concept whose main focus is the relationship with the environment. It searches for solutions that solve the problems of excessive consumption and ephemeral product life cycles. The search for these solutions occurs in the different stages that the product goes through, which can be reflected in principles of reducing the use of material and energy; reduction of waste generated; reduction of energy consumption and harmful emissions in the production process. In addition, Ecodesign also includes collaboration with public and private organisations that can contribute to more environmentally sustainable production (Anicet, Bessa, & Broega, 2017).

Figure 2 shows a schematic diagram that compares circular economy with ecodesign.

Industrial Ecology (IE)

According to (Graedel, 1996) in his article "On the concept of Industrial Ecology", the concept of industrial ecology arose from the idea that industrial activity can be approached or function as a biological ecosystem and that, like a biological ecosystem it would be possible to integrate and balance activities with civilizations and resources. Thus, this same author defines, in another document, the concept of IE as:



Figure 1: Schematic diagram of Circular design.

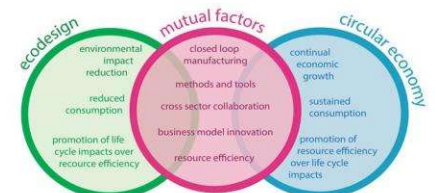


Figure 2: Schematic diagram of the relationship and differences between circular economy and ecodesign.

'(...) study of the means by which Humanity can deliberately and rationally approach and maintain a desirable carrying capacity, given continued economic, cultural, and technological evolution. The concept requires that an industrial system be viewed not in isolation from its surrounding systems, but in concert with them. It is systems view in which one seeks to optimise the total materials cycle from virgin material, to finished material, to component, to product, to obsolete product, and to ultimate disposal.' (Graedel, 1996).

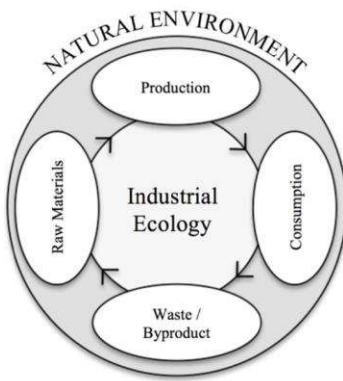


Figure 3: Schematic diagram of the Industrial Ecology concept.

This concept conveys an idea of circularity of materials and encompasses the different stages of production, including those that are somehow involved even if not directly in the production factory. In this concept, it is expected that the way in which budgets are made is considered and the income and outcome of resources are studied, in order to constantly seek how to reduce waste, and even these, how they can be recycled within production itself, or in other companies or sectors. (Figure 3) In short, it studies all the impact that the use of certain resources, by a certain human action, can have on the planet (Graedel, 1996).

Cradle to Cradle (C2C) vs. Cradle to Grave (C2G)

Cradle to Cradle is another one of the concepts often discussed when talking about design strategies for environmental sustainability. This term was created by the designer and architect William McDonough and the chemist Michael Braungart who based themselves on Industrial Ecology to develop it. This model seeks to replace the current model of use and disposal which the creators of C2C refer to as Cradle to Grave given the linear idea of birth and death (Vavolizza, Chaves, & Heemann, 2015).

It is a design and production concept whose objectives are to develop strategies that allow eco-efficiency and lead to zero waste emissions and minimise the negative, but unintentional, consequences of the production processes and consumption of products, considering social and economic aspects as well as environmental ones (Chagas & Caldeira-Pires, 2022). To this end, this model seeks to reduce the extraction of raw materials, analyses the materials and environmental impacts of production processes, and takes into account consumer preferences and their economic situation. Thus, the values associated with this

model are innovation, product quality and the paradigmatic methodology, always with a focus on returning materials to the production cycle as many times as possible (Chagas & Caldeira-Pires, 2022) (Vavolizza, Chaves, & Heemann, 2015).

Upcycling (and downcycling)

Upcycling and downcycling are concepts associated with recycling. The term upcycling was used by William McDonough and Michael Braungart in 2002, and the purpose of this concept is to avoid discarding useful materials (Lucietti, Ramos, Soratto, & Trierweiler, 2017). It is characterised by the process of transforming materials, which until now, were considered obsolete, at the end of their useful life, or waste, into new products. They are used in the state in which they were found, contrary to what happens in the recycling process, downcycling, where products are recycled using chemical and physical processes that, in addition to consuming more energy, usually result in a product with inferior value and performance. This occurs because the integrity of the material is compromised in some way. In upcycling, products maintain or increase value and quality (Figure 4) (Anicet, Bessa, & Broega, 2017) (Moreira, Marinho, Barbosa, & Bizarria, 2018).

Thus, through upcycling, the waste of materials and the excessive use of virgin material are avoided and excess production, energy consumption, air and water pollution and even gas emissions into the atmosphere are reduced. It has become one of the preferred concepts in industries because, in addition to having environmental benefits, it also reflects a reduction in costs (Anicet, Bessa, & Broega, 2017) (Lucietti, Ramos, Soratto, & Trierweiler, 2017).

2.3. The world's use of resources and waste generation and management

Nowadays, humanity has been using resources like no other generation. The use of natural resources has been spent 1.7 times faster than what the ecosystem can regenerate (Ribeiro, 2020). Worse than this, the levels of consumption are directly associated with population growth and its purchasing power which leads to an increase of production and, consequently, an increase on the waste generation (Provin, Dutra, de Sousa e Silva Gouveia, & Cubas, 2021). This means that more and more waste is thrown away, when industries could actually be using it

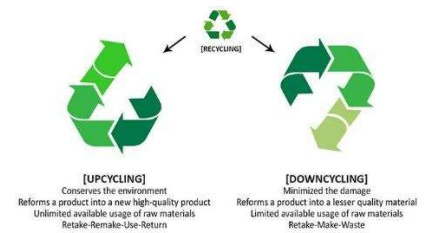


Figure 4: Upcycling, Recycling, and Downcycling symbols.

as the valuable resource it can be. As the earth faces this huge problem, conscious production is no longer a choice, but rather a necessity. People should be rethinking the way to use raw materials and try to find solutions that favour the recycling of materials.

With this in mind, the need to study waste management and the life cycle of products has been growing and, obviously, as shown before the circular economy concept has been playing the biggest role.

One of the most recent strategies developed in Europe to tackle the environmental problem the world is facing was presented in March 2020 by the European Commission, and it is called The Circular Economy Action Plan which is a strategy to a more sustainable and circular economy within the European Union (EU). As said before, the EU has been implementing measures of circular economy since 2015 but the plan has been evolving over the years. This plan presents a set of initiatives and measures regarding design for circularity, sustainable consumption and production, waste management and recycling, as well as many other relevant topics (European Commission, 2020). This plan also identifies seven production chains that require the urgent use of these measures and initiatives, one of them being the Textiles Industry (Vidaurre-Arbizu, Pérez-Bou, Zuazua-Ros, & Martín-Gómez, 2021).

The best way to explore the improvement of waste management is through partnerships between sectors since one's waste can be the production's main resource of another (Provin, Dutra, de Sousa e Silva Gouveia, & Cubas, 2021).

Studies and the search on developing innovative materials that repurpose waste products have been on the front line of new materials.

Actually, according to (Vidaurre-Arbizu, Pérez-Bou, Zuazua-Ros, & Martín-Gómez, 2021):

'The trend in exploring innovative circular materials derived from diverse residues, but mainly from those of organic origin, receives increasing support from public agencies, governments and policy makers as a solution to the environmental problem caused by the linearity of the majority of production processes.' (Vidaurre-Arbizu, Pérez-Bou, Zuazua-Ros, & Martín-Gómez, 2021).

2.4. Wood Waste in Chile and Portugal

In Chile, in the last few years, the total waste production has seen an increase of 28% due to economic activity growth, that translates to more than 17 million tons per year in which 10.4 million of these are industrial related (Alarcón, et al., 2022).

Approximately 5 million tons of wood waste are generated annually in the country, according to the National Forestry Corporation (CONAF), of which 70% originate in the forestry/transformation industry and the remaining 30% are from urban and rural activities such as construction, demolition, maintenance of parks and gardens, ... (Forestal, 2023).

This translates in the transformation sector being the second largest waste generator with 18% of the total in Chile.

The main use of Chilean wood is for internal consumption and mainly used in the cellulose and sawmill industry, production of sheets and veneers, furniture, and other wooden artefacts.

The waste generated in this sector includes bark, sawdust, wood chips and shavings which have many different destinations that can be improved to create more value. For example, plywood manufacturing generates between 40% to 55% of wood waste that could be reused in products with great value (Alarcón, et al., 2022) (Vidal, Balocchi, & Fuentealba, 2021).

The Chilean wood sector already makes an effort to reuse some of the waste as an energy source, to create wooden pellets, some reintegrate the industry in the wooden panels and sheets manufacturing, but it is only between 5 to 20% of the waste that is actually used (Alarcón, et al., 2022).

Regarding the Portuguese territory, according to the National Forestry Inventory of Portugal (IFN), the country's production of wood waste is estimated around 5.6 million cubic metres per year. The main generator of this waste is the forestry sector, responsible for 80% of the total and the type of waste is very similar to the Chilean's ((IFN)., 2010).

As in Chile, Portugal is also making efforts to increase the value of wood waste and in recent years there has been an increasing interest in the production of wood pallets using this waste to use as a renewable fuel source (Almeida & Ferreira, 2019).

Since the waste reuse effort has thus far had a small impact, it is important to promote initiatives that research the efficient reuse of these valuable materials in the development of new products, leading to a decrease in the impact these industries have on the environment.

The goal with this project is the development of new products that integrate the solid waste generated by the wood processing industry through design and engineering.

2.5. The textile coating technique

The textile coating industry was born long ago when people discovered that if they smeared animal grease onto skin or a cloth that it would become more waterproof.

Nowadays this industry is part of one of the biggest industries in the world: the textile industry, and a lot of research is being done in this field as it is rapidly growing.

Coating is used for many purposes such as aesthetics, improving the performance of fabrics, and conferring special properties and functionalities.

Coating has been proven to be a very important technique to add value to textiles and is considered a sustainable technology because water, energy and chemical consumption are reduced by its use.

In this chapter this technique is explored in depth and all issues related to it. It's important to understand what it actually is, by using two quotes from two different references, to better describe it:

'(...)coating (...) of textiles are textile finishing processes designed to add or improve function, to add value to a material, and/or to create a material with specific properties.' (Smith, 2010).

'Coating is a process in which a polymeric layer is applied directly to one or both surfaces of the fabric. The polymer coating must adhere to the textile and a blade or similar aperture controls the thickness of the viscous polymer. The coated fabric is heated and the polymer is cured (that is, polymerized).' (Singha, 2012).

This is a rough description of what the coating technique is but there is much more to it: from the formulation to the final product as well as the many products and machines/ methods that you can use in this technique and, the parameters that you have to assess during this process.

2.5.1. The Coating Methods

The formulation of coatings is complex, they can have a large range of different materials in its composition, depending on many factors that go from the type of textile substrate that is being coated, to the type of base material being used to the results and purpose wanted.

The materials used in coatings are mostly in the liquid form, although there are some in the powder state and even in gas state. This is another variant to the way a coating formulation is prepared (Smith, 2010) (Singha, 2012).

Depending also on the result wanted, the formulation of a coating can be in different forms like pastes, foams, and other types.

As already mentioned, there are many variants to prepare a formulation but there is a common objective to any formulation, “producing a uniform and stable coating with the desired thickness, and with good or desired adhesion to the substrate surface.” (This is, of course, if you don’t want an unusual, uneven, not uniform or unstable type of result to actually be your result) (Smith, 2010).

Pretreatment of Textile Substrate

The textile material has a significant impact on the achieved results, so, it is important to make a previous pretreatment which can include different operations, such as washing, bleaching, curing and a special pretreatment to avoid high penetration of the coating to make sure it is homogeneous and that there will be no interference with the coating adhesion to the surface. (Athalye, 2023).

Preparation of the coating formulation

The next step is the preparation of the coating formulation to be applied on the surface of the textile material.

A coating formulation can have in its composition a large range of different products depending on the result wanted and the



Figure 5: (1) Example of products used in a coating formulation and (2) an image of how the paste using these products looks like.

the method of coating that is being used. Most coating formulations include a specific type of binder and the necessary additives to achieve the wanted result (Example in figure 5).

The binder is mostly an organic polymer or a type of polymer that allows the coating to be polymerized. There is a large range of different polymers in the market that are available for coating techniques. The process of choosing, again, depends on the objective of the coating and, sometimes, it is necessary to try more than one to experiment which is the best option to achieve what's needed. The polyurethane binder is the most common used for water-based systems to produce coatings for applications such as fashion pieces, accessories, upholstery, interior auto decoration, etc.

Regarding the additives, depending on what is the goal, there are also a wide range from crosslinkers, thickeners, antioxidants, emulsifiers, pigments, ... and subtypes to many of them, making it necessary to try more than one to achieve the best result. What is important is to make sure that all the ingredients are compatible with one another (Singha, 2012).

The coating materials, as said before, exist in many forms like liquid, powders, films, ... and in some cases they come from the suppliers ready to use and apply, but, in other cases, some come in different parts and have still to be mixed and prepared to integrate the mixture for the coating (Smith, 2010).

To improve some mechanical resistance properties usually a topcoat is applied (a very thin layer without colour) on the top of the coating. This topcoat is usually polyurethane based.

During the preparation of the coating formulation there are many processing parameters that need to be controlled that can affect the result. Some of these were already mentioned above, however, the most important parameter to control when the coating is done, is the viscosity of the paste. The viscosity can affect the transferring and deposition of the coating. It depends on paste to paste, the result required and the substrate whether the paste should be more, or less viscous (Smith, 2010).

Coating application on the textile substrate

Nowadays a coating can be applied to basically any type of textile, in terms of composition as well as its form: it is possible to coat yarns, woven fabrics, knitted fabrics and nonwoven.

For the purpose of this dissertation, it will only be approached the coating of woven and knitted fabrics.

Also, there are many types of coating methods one can choose. Here, as in many other choices regarding coatings, the type you choose depends on the requirement of the end product (Singha, 2012).

Some examples of these types of coatings are knife on air coating, knife-over-roll coating, transfer coating, roll coating, immersion/dip coating, and there are some others (Smith, 2010) (Singha, 2012).

In this project the process of coating used was the knife-over-roll coating. Figure 6 shows a schematic diagram of this process in which the fabric is stretched flat, so it is an even and uniform surface. The knife is suspended above the fabric and the roller is underneath the fabric. The height in which the knife is held above the fabric can be changed and be set very accurately. This allows a gap between the blade and the fabric, and this is how you can control the thickness of the coating layer one can apply (Smith, 2010) (Singha, 2012).

Drying

After the coating application it is necessary to proceed to the drying operation, to evaporate the water of the coating and volatile compounds of the formulation. The drying is usually performed in an oven at around 100°C with hot air.

Curing

Curing is a crucial operation to improve the fixation of the coating to the textile substrate. This process is done with hot air around 140 °C - 150°C for 3 to 5 minutes in an oven (Sen, 2008).

Finishing

After applying the coating layer(s) there are still some finishing processes that can/need to be used that range from chemical to mechanical processes.

An example of a chemical finishing process is the application of topcoat by knife coating process. This can give the product some other desired properties that, with just the coating layer(s), is not possible. These properties can be glossing treatment, softness, resistance to abrasion, among others. These are usually applied before curing.

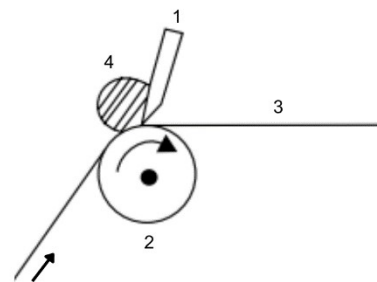


Figure 6: Schematic diagram of the knife-over-roll coating application. (1) knife; (2) roll; (3) substrate; (4) coating material.



Figure 7: Example of an embossing. (1) The pattern used to create the texture on the surface; (2) the coated surface after the embossing process. It is visible where it was applied and where it wasn't.

For the special effects, an example is the embossing process which is a type of calendering. It consists of an engraver that is done on top of the coating or topcoat. There are many types of calendering but in short, it is done by “passing the textile substrate in between two heated drums or rollers” where temperature, and pressure act simultaneously to modify the coating surface. Most types of calenderings have the purpose to smoothen the surface of the product and increase its the mechanical properties. However, the embossing process is used to engrave patterns or designs to the coating surface to obtain different effects as it is possible so see in figure 7 (Rahman, 2023).

This process can be done before or after curing depending on the required final product.

2.6. Polymers and Biopolymers used in the coating industry

A polymer is a molecule composed of many monomers joined together. When regarding polymers for textile coating, these are mostly synthetic meaning they are made from a chemical source (Britannica, 2023). There are many polymers in the market for coating. They can be divided among four categories: rubbers, polyvinyl chloride (PVC), polyurethane (PU) and acrylics. The use of the polymer depends on the desired features wanted in the coating since each polymer can bring different properties such as durability, water resistance, abrasion resistance, etc. These options, as said before, are the most common, but they have a high environmental impact since they depend on non-renewable resources and their degradability is much harder to achieve. (Sen, 2008).

The good news when trying to avoid the use of these synthetic polymers is that there are biopolymers. These are obtained from natural and renewable sources such as plants, animals or microorganisms. For the coating industry the main source of these biopolymers are substances such as starch, chitosan, alginate, or cellulose. The main advantage of the use of these options when coating is that they provide unique properties like biodegradability, biocompatibility and a lower impact when compared to synthetic polymers (Barbosa de Araújo, Arruda, Alves, & Araújo, 2021).

There is also a third category on coating polymers that is somewhat in between the two above mentioned when looking through the environmental impact lens, the bio-based polymers. Although, one may think that biopolymers and bio-based polymers are the same thing, that is not necessarily true. They both have a connection to renewable sources, but they are distinct.

Bio-based coating polymers are indeed derived from renewable sources. These are produced using bio-based monomers retrieved from renewable sources. Some examples of bio-based coating polymers are bio-based PU, bio-based polyethylene, and bio-based polyesters.

Some bio-based polymers can be considered biopolymers if they are obtained from natural sources but not all of them are. The distinction between the two is in the origin and composition of them. Bio-based polymers production focuses on the renewable property of the source while biopolymers are more focused on their natural origin which mostly assigns them biodegradable and biocompatible properties (Ribeiro, 2020) (Commission, Bio-based products, 2023).

Bio-based polymers, with regards to sustainability, may not be the best option, but sometimes, and mostly due to the desired properties wanted with the coating, the use of biopolymers is not possible so, bio-based polymers are a viable alternative to reduce the use of fossil-based options like shown above.

Figure 8 shows a schematic diagram that summarises the differences between synthetic polymers, biopolymers, and bio-based polymers. (Ribeiro, 2020)

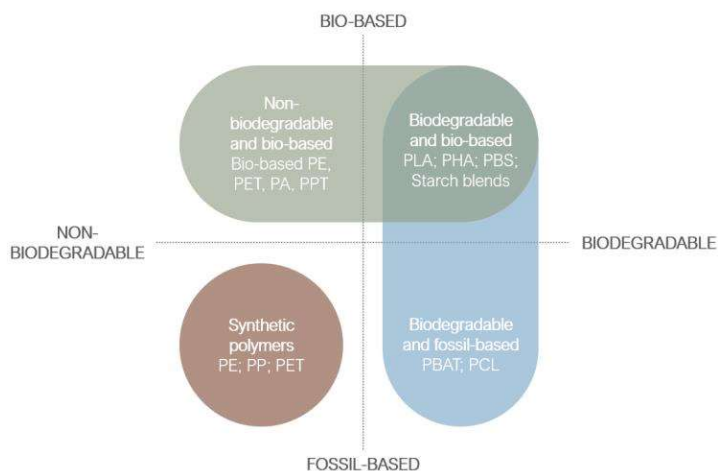


Figure 8: Schematic explanation of the difference between synthetic polymers; bio-based polymers and biopolymers with examples of each.

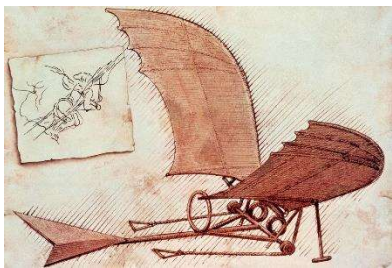
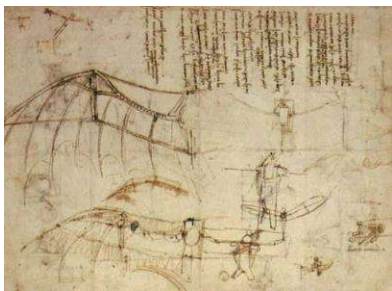
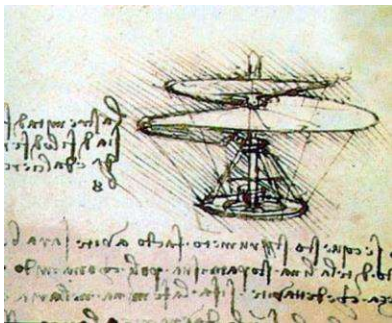
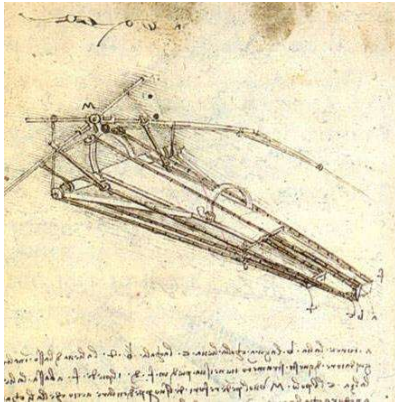


Figure 9: Leonardo Da Vinci's drawings on flying mechanisms.



Figure 10: Example Leather alternative made from pineapple leaves.

2.7. Biomimetics

Biomimetics is a term that comes from the Greek words 'bios' that means life and 'mimesis' which means to mimic, so, biomimetics basically means mimicking biology or nature.

The word first appearance was in the Webster's dictionary in 1974 and was defined as:

'the study of the formation, structure or function of biologically produced substances and materials (as enzymes or silk) and biological mechanisms and processes (as protein synthesis or photosynthesis) especially for the purpose of synthesising similar products by artificial mechanisms which mimic natural ones.' (Bhushan, 2009).

It is a strategy whose purpose is generating sustainable solutions that one can apply when developing materials, products, or processes. Design that is inspired in biology or derived from nature is design that applied principles of biomimetics (Barbosa de Araújo, Arruda, Alves, & Araújo, 2021).

A great example of someone who applied biomimetics in his creations was the genius Leonardo Da Vinci who in 1488 invented the first flying mechanism based on studies of how birds fly (Figure 9) (Bhushan, 2009).

Biomimetics principles can be applied to basically any field. These principles have been being applied in the textile industry for many years and for many purposes. One big example, and within the scope of this dissertation, is the continuous research that has been carried out to try and create materials that can mimic leather (Example in figure 10), some of which will be discussed next.

2.8. Active, living, and adaptive materials

According to (Alarcón, et al., 2022), the responsible investigator of the project of which this dissertation is a part, there should be a base for the designing of the projects that integrate this research, on active, living, and adaptive materials. The investigator defines these materials approach as:

*'(...)the approach of active, living and adaptive materials, which, of biological, chemical or algorithmic origin, constitute a different way of understanding and mobilising materials in design.'*¹ (Alarcón, et al., 2022).

¹Citation translated by the dissertation author from the original in Spanish: "el enfoque de materiales activos, vivos y adaptativos, los que, de origen biológico, químico o algorítmico, constituyen una forma diferente de comprensión y movilización de los materiales en diseño."

According to this investigator, active materials are the ones that react to external agents, while the living materials are the ones that can grow or rearrange, or transform autonomously, and the adaptive materials define a new condition to prime matters by reinterpret, reuse, or reinvent resources. The focus on this way of developing materials is the experimental knowledge that design, and engineering are based on, resulting in new and innovative materials.

The role of designers and engineers in developing these innovative materials is essential to research, discover, experiment, and develop these new possible materials instead of simply using the existing ones that may be harming the environment or are simply inefficient. The development of new materials is an emerging area and technologies, strategies, and tools, need also to grow and adapt to these new developments in order to meet the needs and generate changed growth and improve the world's answer to environmental problems (Alarcón, et al., 2022).

2.9. The leather industry

The leather industry is in a crisis, thus there is an increase in interest in finding new and more sustainable leather alternatives, not only among manufacturers that are aiming to meet society needs and demands, as well as governmental measures and laws, but also among customers that are beginning to understand the problem and its urgency and are pressuring the industry (Spinnewijn, 2022).

Some manufacturers assert that leather is already a subproduct from the meat industry, that it is already part of the circular economy of meat production, so it is sustainable, however, leather has three times more environmental impact than its synthetic alternatives made with PU (polyurethane) (Ribeiro, 2020).

Although this is true, leather is still considered a very important material due to its unique properties.

These properties have been the biggest struggle for the industries of alternative materials to leather which have been continuously researching these features to achieve similar performance on these new products.

2.10. Alternatives to Leather

The first alternatives to leather were created, not because of environmental worries, but due to monetary ones. Leather has always been a very expensive material, so the first alternatives emerged as a way to have more accessible leather like materials in the market (Ribeiro, 2020).

The first options to be available were synthetic, fossil based but nowadays, the focus is mainly in finding materials that are environmentally more responsible, so, bio-based options have been emerging and these more sustainable options are getting wider and more differentiated.

2.10.1. Synthetic Leather

As said above, nowadays the options to replace leather are numerous, some more polluting than others. The chemically produced ones have been the most popular. These options have many designations in the market like artificial leather, synthetic leather, faux leather, pleather, among others. In this document it will be referred to as synthetic leather.

These materials are produced with two or more layers of polymers like PU or PVC, coating some type of textile substrate (Ribeiro, 2020) (Meyer, Dietrich, Schulz, & Mondschein, 2021).

Synthetic leather is considered a polluting product due to being fossil-based and consequently not biodegradable, and due to its manufacturing process waste and contamination of water, and incorporation or use of chemicals and other materials that are also noxious to the environment (Ribeiro, 2020).

2.10.2. Bio- Based Leather Alternatives

Bio based materials are materials that are in its whole or in part “derived from materials of biological origin, excluding materials embedded in geological formations and/or fossilised.” (Commission, Bio-based products, 2023).

These can be plants, agriculture wastes, marine matter, forestry derived, ...

The advantages of using bio-based products are numerous, from decreasing water and energy consumption in the production, reducing toxic waste, reducing the CO₂ emissions, among other advantages (Commission, Bio-based products, 2023).

Regarding the bio-based alternatives to leather, as said before, more and more options are becoming available. Some examples of these options are leather like materials derived from pineapple fibres (Piñatex®); derived from mango (Fruitleather Rotterdam); a type of mushroom; cactus leaves (Desserto); apple wastes (Appleskin); Kombucha, ... These are the examples that are going to be presented in this dissertation. These alternatives emerged due to a change to a more conscious behaviour and perception of the consumers. The acknowledgement that the use of plastics was and is getting out of control and the impact of this industry on the environment, was one of the main reasons for the increased interest in developing these new materials (Ribeiro, 2020).

The processes to create these new alternatives are, as the options of materials, numerous and different from each other. These processes are not all impactless in the environment. The objective is always to try and achieve an impactless process of production, however, that is not always possible. Despite this, these processes are indeed more sustainable and less harmful to the environment (Ribeiro, 2020).

2.10.2.1. Pineapple leaves leather alternative

Piñatex® is one of the leading plant-based leather alternatives. This material is made from fibres that are in pineapple leaves.

To produce this material, the fibres from the pineapple leaves are extracted from them and then dried naturally in the sun. After this process they are mixed with polylactic acid (PLA) obtained from corn starch and a non-woven mesh is created with this mixture. To finalise the production process this mesh is then coated and becomes the final product (Spinnewijn, 2022).

This product was developed by Dr. Carmen Hijosa who also is the founder of Ananas Anam, the brand that sells products using the material she created.



Figure 11: Hugo Boss Shoes line made with Piñatex®.

Piñatex®, uses as its raw material waste from the pineapple industry, creating a circular economy relation within these two industries.

This material can be used to create fashion products, accessories, and upholstery. The brand states that more than 1000 brands from all over the world have already used this material and among these brands are Hugo Boss as seen in figure 11, H&M and the Hilton Hotel Bankside (Anam, 2023).

2.10.2.2. Mango-based leather alternative

The brand Fruitleather Rotterdam created a material using waste from the mango industry. This material is created using the pulp of mangos that were considered not fit to be sold and would be sent to trash. This pulp is then mixed with some binding agents resulting in a pure like mixture that is poured on trays and heated in an oven. The sheets of material are then retrieved from the oven and coated with a leather finish to make the material more durable (Spinnewijn, 2022) (Harger, 2023).

This product is produced in trays that are not very big, which means that the resulting sheets allow only to create certain products that don't require large amounts of textile, like shoes, handbags, and wallets (Harger, 2023). An example is the key chain in figure 12.

Fruitleather Rotterdam collaborates with brands to deliver the material for their designs.

Much like Piñatex®, Fruitleather uses a by-product of the mango industry which creates a circular economy relation between these two industries.



Figure 12: Fruitleather x Windmilkey collaboration.

2.10.2.3. Mushroom made leather alternative

Another alternative being developed is a mushroom based leather alternative. In this case, many researchers and companies are developing this type of material, for example, MuSkin®, Mycotech and Mylo™.

This type of material is produced using a type of mushroom called Mycelium.

Mycelium grows underground and it is composed by a network of roots, called hyphae, looking like threads that branch and fuse together, creating the network. This network can span for kilometres so that Mycelium are considered the largest organisms on earth (Khurana, 2021) (Spinnewijn, 2022).

The details on how the mycelium-based leather is produced are different between companies and researchers, and since it is an innovative material, a lot of information on its production is kept secret, however, the main process outline is retrievable. To obtain this leather alternative, fungi spores are combined with previously treated sawdust and other organic materials, and this mixture is set in an environment with high levels of CO₂ to grow while the temperature and humidity are kept in very tight control - as these are the most influential factors on the mycelium growth. The mycelium takes about a week to grow into a foam-like structure, and this is what is used to create the leather alternative. This structure is then washed, treated and after that, some agents are added to improve the mechanical properties of this material. Next, it is compressed, and a moisturiser is added to assure the material keeps its flexibility. After this, it is ready to be dyed and embossed (Spinnewijn, 2022).

Pure mycelium is biodegradable but due to the use of chemicals in the treatment steps, this leather alternative degradability varies, and, in some versions, plastic is added. It is not the perfect solution, but it is indeed a more sustainable option than leather itself or synthetic leather.

This material can be used in a large range of products including shoes and accessories. The mycelium product Mylo™ from Bolt Threads partnered up with the global leader in sportswear Adidas and in 2021 launched a conceptual version of the iconic Adidas Stan Smith shoes made with the mycelium-based leather alternative Mylo™ (Figure 13). It is the first shoe ever made with a mycelium-based material (Mylo™, 2023).

2.10.2.4. Cactus-based leather alternative

One other leather alternative is made using cactus leaves. This material is called Desserto® and was conceived by the company Adriano Di Marti.

To produce this material, the cactus leaves are harvested, cleaned, and are dried for three days in the sun.



Figure 13: Mylo™ x Adidas Stan Smith made with mycelium-based leather alternative.



Figure 14: Karl Lagerfeld X Amber Valletta handbag made with Desserto® leather alternative.

After this, the fibres and proteins of the leaves are extracted and mixed with a polymer compound and applied in a woven basis of polyester cotton or both, depending on the end product.

As similar as the other bio-based leather alternatives presented before, Desserto® is less impactful when compared to traditional and synthetic leather (Spinnewijn, 2022).

Desserto® material has been used by many brands, one of which is Karl Lagerfeld. This brand used Desserto® in many products like handbags (example in figure 14), shoes and “leather” jackets in the two capsule collections Karl Lagerfeld X Amber Valletta launched in 2021 and 2022 (Intravaia, 2022).

2.10.2.5. Apple based leather alternative

AppleSkin™ is a material created by the Italian company Mabel and it has in its composition apple waste from the beverage industry such as cider and juice.

The production of this material is very similar to the Desserto® production. Apple pomace is collected from the beverage industry and then washed and dried. After this process it is converted into a powder that will then be added to a water-based polyurethane creating a mixture that is used to coat a lyocell base made with cellulose extracted from wood pulp (Spinnewijn, 2022) (AppleSkin, 2023).

This material, as the ones presented above, can be used for many purposes such as bags, shoes, upholstery, ... an example is the Black Gala Backpack (Figure 15) from the brand Allégorie which is a brand that only uses eco-friendly materials in its products.



Figure 15: Black Gala Backpack from Allégorie made with AppleSkin™ leather alternative.

2.10.3. Lab-cultured leather

One of the most recent attempts to create a leather alternative is the lab-cultured, lab-grown leather. Two companies have already patented technologies to manufacture leather in laboratories.

One of the companies researching this type of materials is the New Jersian Modern Meadow, they patented a material that uses yeast cells that are fermented in a process similar to brewing beer to obtain collagen (Figure 16).



Figure 16: Modern Meadow's Lab-Cultured Leather.

The resulting collagen is a liquid that can be arranged in any shape or used to bond fabrics. To create sheets looking like leather, the company adds enzymes to alter the structure of collagen. After this process the result is a solid and fibrous material. At the moment this material is still on the lab scale, but Modern Meadow has already partnered with many companies and industries to try to elevate it to industrial production (TONDO, 2021).

VitroLabs Inc. is the other company, located in San Francisco that is exploring tissue engineering to produce a leather similar option using the animal cells. To do so, the cells are extracted harmlessly from the animals and then multiplied in laboratory and set in a prosperous environment for collagen production. A specialised bioreactor is used to help the cells grow and form into tissue. The result is leather like tissue that undergoes a tanning process after (Figure 17).

Although this process uses cells from the animals, the process of developing the cells in laboratories takes weeks, while growing the animals to extract traditional leather takes years and billions of gallons of water along with many other environmental issues, which represents a significant reduction in environmental impact for VitroLabs Inc..

The material obtained by VitroLabs Inc. is also still in lab scale production, but it is a very promising material as its complexity and quality is similar to the traditional leather (TONDO, 2021).

2.11. Chapter's summary

To sum up, design and designers have an important role in sustainability, since it is in the design process of objects that the environmental thinking should begin. To do so, many strategies began emerging to help develop these more responsible products.

Circular design and circular economy are the main models to most sustainable strategies developed. Circular Economy takes in account the consumption and waste of resources and studies the optimization and management of these in order to reduce the use of virgin materials and give value to waste. Circular design seeks to change the current conception of production to a more responsible, informed, conscious and sustainable one.



Figure 17: VitroLabs Inc. Lab-Cultured Leather.

The strategies that involve these models are emerging. Many of them exist now and are a fitting response to the urgent environmental problem the world is facing.

Humanity has been using resources like no other generation due to the hulking consumption mentality that, associated with population growth and purchasing power, leads to an increase in waste generation. This accentuates the need for a change in mentality and the study and optimization of waste management. The best way to explore the improvement of waste management is actually through partnerships between sectors since one's waste can be another's main resource.

Wood waste is a great issue in Chile and in Portugal, so it is important to promote initiatives that research the efficient use of these wastes and integrate them in new products through design and engineering.

This chapter also explains the coating technique and polymers since the scope of this dissertation involves the use of wood waste in the textile industry through the coating process to develop a material that can be an alternative to leather.

Continuous research on the development of materials that can mimic leather have been carried out and are emerging quickly in the market. This type of material development involves principles of biomimetics, which is the study of something that exists in nature or that nature creates and try to mimic it or its properties to reduce the use and consumption of the materials that are being mimicked.

The development of leather alternatives is indeed important since it is a very harmful industry for the environment due to the use of chemicals to treat the leather, even though many manufactures assert that leather is already a subproduct from the meat industry, basically meaning it already applies circular economy in its production, In fact, it can be three times more environmentally impactful than its synthetic alternatives made with PU, which are not the best alternative either.

The synthetic alternatives are fossil based and were the first alternative to be available, not due to environmental concerns but due to monetary ones. They were created as a cheaper and more accessible alternative to leather. Nowadays the focus is mainly in finding materials that are environmentally more responsible, so, bio-based options have been emerging and these more sustainable options are getting wider and more differentiated.

3. DESIGN OF THE COATED FABRICS USING PINE WOOD WASTES

3.1. Introduction

The main goal of this dissertation was to design a more sustainable coated fabric, using pine wood waste generated in the wood transformation industry. This would be the beginning of an experimental textile coated material that could become a viable alternative to leather.

This dissertation is part of an international project, and it required the use of pine wood waste from Chile, but since this project was developed in Portugal, Portuguese pine wood waste was also experimented with.

In this chapter all the experimental parts of the development of the coated fabrics will be presented: the one using the Portuguese pine wood waste (pine bark) and another one that uses the Chilean pine wood waste (pine sawdust).

First, the preparation steps necessary, before the coating experiments, like the drying of the pine bark and the cut milling of the pine bark and sawdust, will be presented. The process of ball milling of the pine bark will also be presented since, in some of the tests the waste was milled through that process.

Secondly, a template description of the steps usually followed in the experiments to prepare the coating formulation and application is presented.

In the end of the chapter a detailed description of the considered final solutions is presented while the details to all the other experiments done to reach these solutions are in the appendix section of this dissertation.

Figure 18 presents an overall schematic diagram of the process usually followed throughout the experiments done.

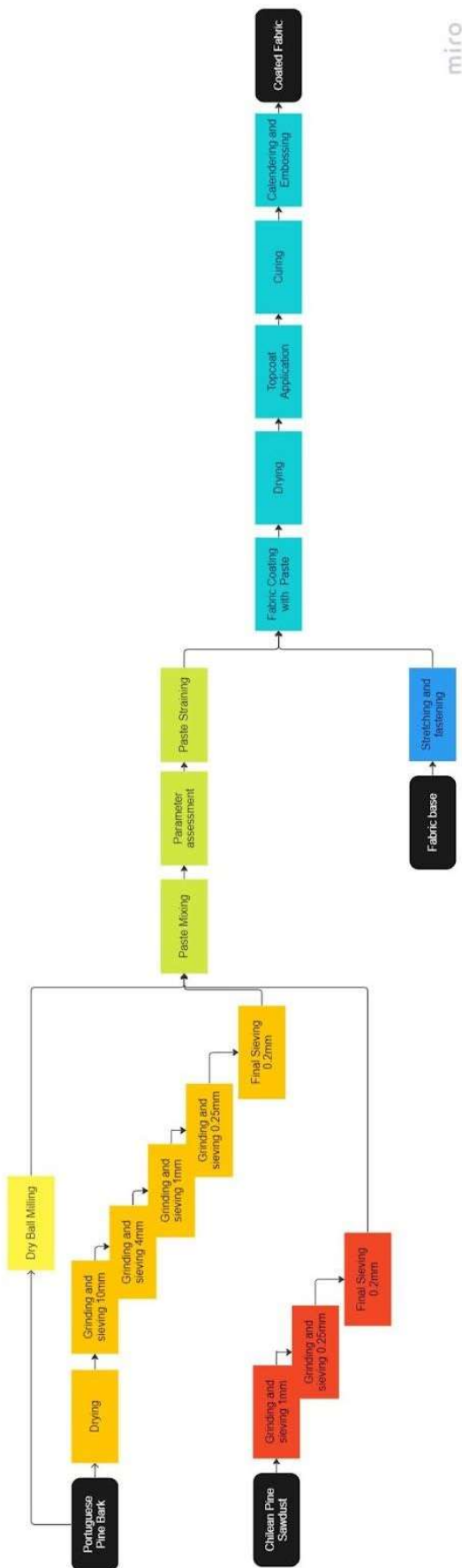


Figure 18: Schematic diagram of the steps of the Material Development.

3.2. Previous steps to coating

3.2.1. Drying the pine bark

One type of waste used in the experiment was pine bark from Portuguese pine trees.

This waste was delivered in its original form, only broken in pieces.

Experiments with this waste were done both using it without previously drying and some other tests using it after being dried.

To do so, these pieces of pine bark were broken into smaller pieces sized around 5 x 5 cm and placed in a tray. This tray was inserted in a drying tunnel (figure 19). The pine bark stayed inside this tunnel, drying for 26 hours at 60°C.

It was not necessary to dry the Chilean pine sawdust.



Figure 19: Drying and curing tunnel.

3.2.2. Grinding and sieving the pine bark

In order to use this waste in a coating formulation it was required for it to be in a powder state so, it was necessary to shred the pine bark. Afterwards, both dried and not dried pine bark were milled, using a cutting mill, which is shown in figure 20. This machine is suited for grinding many types of materials and allows the grinding of these materials in many different sizes, ranging from 0.25 to 20 mm. These sizes are obtained through the combined work of a 6-disc rotor and the bottom sieves that exist with trapezoid or square holes in different sizes (Figure 21 shows some examples).

To mill the pine bark, it was necessary to do it in four different stages since it is not possible to grind the bark with the smallest hole size sieve. It was necessary to do it changing in between four-hole size sieves. The first with square holes of 10 mm, the next, with also square holes with 4 mm, another with, trapezoid holes with 1mm and the last one also a trapezoid hole sieve but with 0.25mm hole size. Figure 21 shows the four sieves used.



Figure 20: Retsch SM 300 Cutting mill machine.

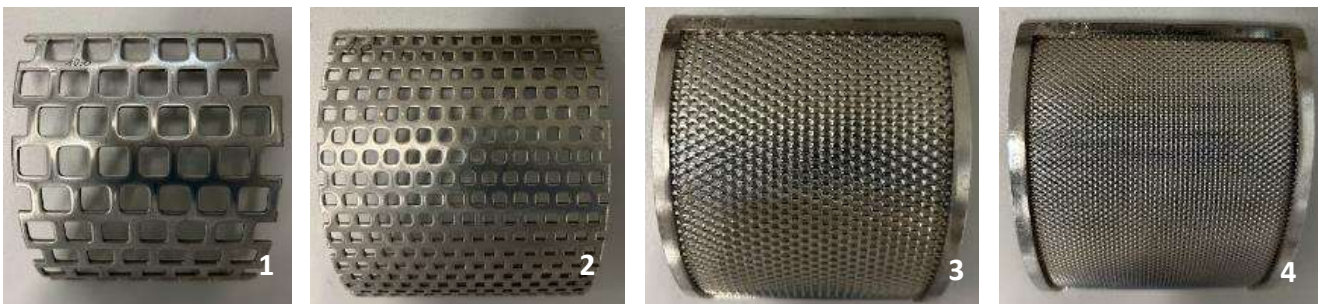


Figure 21: Bottom sieves for the cutting mill; (1) Sieve with square holes with 10 mm; (2) Sieve with square holes with 4 mm; (3) Sieve with trapezoid holes with 1 mm; (4) Sieve with trapezoid holes with 0.25 mm.

The grinding was done at a 1500 rpm speed. The initial state of the pine bark and the sizes of particles after each used sieve are shown in figure 22.



Figure 22: Pine bark throughout the different stages of the cut milling process; (1) initial state; (2) after milling with the sieve with 10 mm holes; (3) after milling with the sieve with 4 mm holes; (4) after milling with the sieve with 1 mm holes; (5) after milling with the sieve with 0.25 mm holes.

3.2.3. Grinding the pine sawdust

The pine sawdust went through the same milling process. Although, not as many grinding stages were necessary since it was presented in a smaller size than the pine bark. In this case, it was only necessary to use two sizes of sieves, the one with the trapezoid hole size of 1mm and the other trapezoid hole sieve with 0.25 mm hole size.

As it was done in the process of milling the pine bark, it was used at a 1500 rpm speed. The initial state of the pine bark and the sizes of particles after each used sieve are shown below in figure 23.



Figure 23: Pine sawdust throughout the different stages of the cut milling process; (1) initial state; (2) after milling with the sieve with 1 mm holes; (3) after milling with the sieve with 0.25 mm holes.



Figure 24: Different views of the Manual sieves used to achieve the 0.2mm particle size.



Figure 25: Retsch PM 100 Ball milling machine.

² These intervals are set for lowering the temperature inside the jar and for the particles to set down.

3.2.4. Sift the pine bark and sawdust powders

After the processes described in 3.2.2. and 3.2.3., it was still required for the waste powders to acquire a smaller particle size. To do so, it was necessary to sift the powders manually through a sieve as presented in figure 24. This sieve allows one to achieve a particle size of 0.2 mm (200 μ m).

Figure 26 shows the waste after this procedure and ready to be incorporated in the coating process.



Figure 26: (1) Pine bark and (2) sawdust particle size after the manual sift process.

3.2.5. Ball milling the pine bark

A side experiment was conducted in which the pine bark waste was milled with another process, the ball milling. As the name says, this process uses small balls to mill the wastes. It is a more complex process and time consuming, but it delivers a smaller particle size. The machine used for this process is shown in figure 25. This machine grinds materials in dry and wet conditions and it is possible to use balls made from many different materials that can range in size from 5 to 40 mm of diameter.

In this case the milling occurred in dry condition and the balls used were ceramic and had 5mm in diameter. The waste used in this process was already previously grinded in the cutting mill. Some of the waste was put inside the machine's jar with the balls mentioned and the parameters it was set for were: 30 minutes of milling time, 1 minute at a time with an interval² of 10 seconds between each minute. The jar rotating direction changed after each interval. The rotation speed was set for 400 rpm.

The sawdust waste did not go through this process since the results in the coating (which will be presented further)

using the waste that went through this procedure were not satisfactory. It was considered an unuseful process for this project's purpose.

Figure 27 shows the result of the ball milling on the pine bark waste.



Figure 27: Pine bark particle size after the ball milling process

3.3. Coating formulation and application

3.3.1. Formulations

The first step to make a formulation paste for a coating is to gather the materials that will incorporate that paste. This can range from polymers to additives and in this case waste products too. The following tables 1 and 2 show all the ingredients used throughout all the formulations experimented.

Table 1: Polymeric components that were used in the coating formulations.

PRODUCT'S GIVEN CODE	CHEMICAL BASIS	DESCRIPTION	TYPE OF PRODUCT	NOTES
POLYMER 1	Water soluble polyurethane	Nonionic thickener of aqueous dispersions of polyurethanes, acrylics, vinyl acetates and emulsion polymers in general	Thickener	Used in combination with TOPCOAT 1
EMULSIFIER 1	Arylpolyglycol ether	Emulsifier and dispersing agent for textile printing	Emulsifier	
CROSSLINKER 1	Waterbased aliphatic blocked polyisocyanate.	Crosslinker with improved reactivity and thermostability	Crosslinker	
POLYMER 2	Anionic, aliphatic polyester-polyurethane	White low-viscosity dispersion in water with a solids content of approx. 50 %	Polymer	
TOPCOAT 1	Water-borne blend of synthetic polymers	Matt topcoat, NEP free, for synthetic leather	Topcoat	Used in combination with POLYMER 1
TOPCOAT 2	Waterborne polyurethane dispersion.	Low gloss coating for textile and synthetic materials. Made with raw materials from vegetal sources, obtained from plant-derived substances. Bio-based content 60%.	Topcoat	
CROSSLINKER 2	Based on polyisocyanate, blocked, free from butanone oxime	formaldehyde-free, polyfunctional crosslinking agent	Crosslinker	
POLYMER 3	Aliphatic polyurethane dispersion	PU dispersion which is stable to hydrolysis, based on renewable raw materials (has a biobased content of 43 %, based on active content)	Polymer	
POLYMER 4	Aliphatic polyurethane dispersion	PU dispersion which is stable to hydrolysis, based on renewable raw materials (has a biobased content of 38%, based on active content)	Polymer	
POLYMER 5	Dispersion of aliphatic polyester-polyurethane	anionic with solid content of 50%	Polymer	
THIECKENER 1	Polycrylate	Synthetic thickening agent for coating pastes	Thickener	
THICKENER 2	Associative PU thickner	Rheological additive for coating pastes	Thickener	
THIECKENER 3	Polycrylic acid	Synthetic thickener for coating pastes	Thickener	
THIECKENER 4	Polyurethane in aqueous solution.	Rheology modifier for waterbased resins system. Bio based content calculated on 20%	Thickener	

Table 2: Other components that were used in the coating formulations.

WASTE TYPE	INITIAL STATE	PREVIOUSLY DRIED/ NOT DRIED	TYPE OF GRINDING
PINE	Bark	Not dried	Cut milled
	Bark	Dried	Cut milled
	Bark	Dried	Ball milled
	Sawdust	Not specified	Cut milled
Distiled Water	Does not apply	Does not apply	Does not apply



Figure 28: Precision scale used for measuring the formulations components.



Figure 29: Techmatic S2 Mechanical Agitator.



Figure 30: Brookfield DVE Viscometer.

3.3.2. Weight measurements

After gathering all the products that will be in the formulation paste for the coating, it is necessary to weight them according to the percentage of each component that the paste should have.

These measurements were done in a precision lab scale, as the one shown in figure 28, from the brand Mettler Toledo the model PG5002-S DeltaRange that measures weights between 0.01g and 5100,00g.

3.3.3. Mixing components

After measuring the products that will be incorporated in the formulation paste for coating it is necessary to mix them. It depends on the components which ones will be mixed first. Later in this chapter it will be described the different experiments done and, in this description, it will detail the order of mixing of the components of said formulation.

This mixing is done using a mechanical agitator and the one available in CITEVE was the Techmatic S2, shown in figure 29.

3.3.4. Measurement of viscosity (and pH)

When the formulation is all mixed the most important information to have of the paste is its viscosity. The viscosity measurement is what allows to know if it is viscous enough to make a viable coating. To measure the viscosity of the formulations a Brookfield DVE viscometer as shown in figure 30 was used.

The procedure to measure the viscosity begins with choosing the spindle. The spindle is a tool that is attached to the viscometer,

put inside the formulation, and it's what will provide the viscosity data. It is a tool that resembles a steel stick with (or without) a disc in one of its ends. There are different sizes of spindles (Figure 32). In this viscometer's case, the spindle sizes are numbered from 01 to 07. The choice of spindle depends on the paste state and should be chosen in order to obtain a torque between 10% and 100% according to the instructions of the viscometer available in CITEVE. The thicker the mixture is, the lower the number of spindle should be chosen. The spindles that were used in the scope of this dissertation were number 05 and 06, being the most used, the number 06.

To have a correct reading of the viscosity, the paste should submerge the spindle until the indentation that each spindle has (see example in figure 33).

The next step is to set up the viscometer for the analysis. After turning on the machine, it is necessary to identify the spindle number and the velocity desired. The velocities range from 0 to 100 rpm and should be adjusted to achieve a torque between the values referred above. The most common velocities used to measure the viscosity of the formulations done in this project were 50, 60 and 100 rpm. After setting up, the motor should be turned on to start the analysis. The display shows the viscosity data, in Pascal Second (Pa.s), the velocity, the torque and the spindle number. Figure 31 shows an example of a measurement in the screen and the buttons to set up the viscometer.

Throughout the experiments and based also in the CITEVE's technician experience, it was concluded that the viscosity of the formulations for this project should be around 4 to 9 Pa.s.

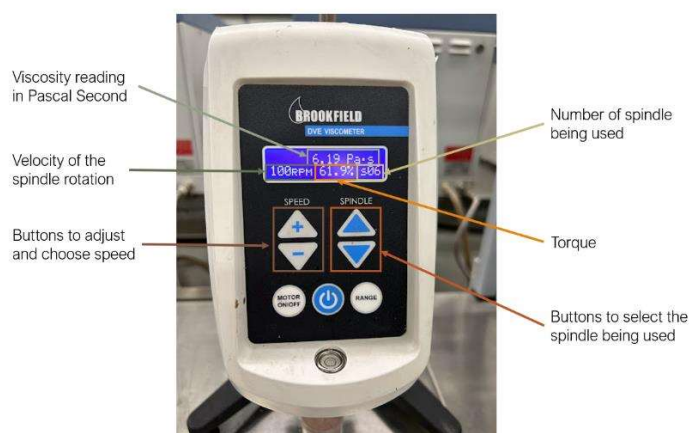


Figure 31: Viscometer display with the indications of its values and function buttons.



Figure 32: Spindle set available for the viscometer used.

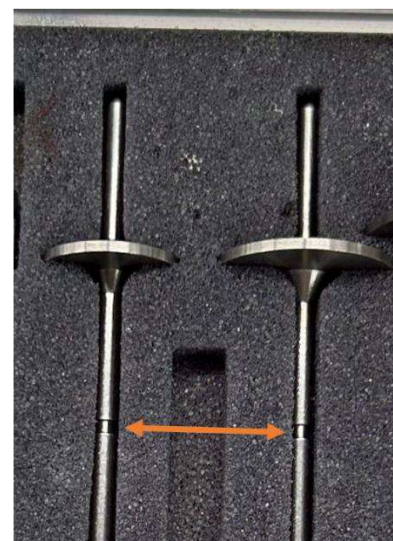


Figure 33: Example of spindle points with the indication of the indentation.



Figure 34: pH equipment, WTW Inolab pH7110 pH Meter.

The pH of the formulation is also usually account for but in case of this dissertation, it didn't interfere or was necessary to measure every time. The instrument used for that purpose was the one presented in figure 34, WTW Inolab pH7110 pH Meter.

3.3.5. Strain the formulation

In some cases, it was necessary to strain the formulations to reduce the lumps of waste in it. To do so, a common strainer was used. Sometimes the paste would be strained more than once. Figure 35 shows an example.



Figure 35: Step of straining a formulation.

3.3.6. Fabric fastening and knife adjustments

When the formulation is done, it is necessary to prepare the fabric and fasten it to the machine so that it is ready for the application.

It is required a piece of about 35 x 35 cm of the desired substrate. It is fastened to the machine in which the coating will be applied. In this dissertation's case, as said before, the method that was used was knife on roll and the machine used was the LABCOATER type «LTE-S» from Mathis AG as shown in figure 36.

The fabric needs to be very well stretched and without imperfections in its surface. Next, it is necessary to position the knife and adjust it to the desired height, which will be responsible for the thickness of the layers. The layer thickness depends on the result wanted.

Figure 37 shows a picture of a fabric and knife prepared for application, indicating the regulators of the knife's height.



Figure 36: Mathis AG LABCOATER type «LTE-S», coating application, drying and curing machine.



Figure 37: Fabric and knife prepared for coating, indicating the regulators of the knife's height.

3.3.7. Textile Coating Operation

The next step is to apply the coating formulation on the textile substrate. To do so, in this knife on roll method, the knife begins being positioned in the fabrics end, closest to the machine. The formulation is then poured near the knife at its whole length as shown in figure 39 (1). Then, the knife and roll are pulled away from the machine to the other end of the fabric and the paste is spread on the fabric's surface (see figure 39 (2)). After this, the knife is removed and washed, the roll is lowered, and the fabric is inserted in the machine for drying. This process can be repeated if more than one layer of coating is desired.

3.3.8. Drying

After the formulation is spread in the fabric it is necessary to dry this layer. The machine is previously set in the desired parameters. In this dissertation's case, the drying occurred always at 100°C air temperature, for 5 minutes and only the fan speed varied between 1300 and 2300 rpm. Later in this chapter the experiments done will be presented and the specifications for each one will be detailed there.

In figure 38 it is possible to see an example of one of the settings of a drying process of a coating application.

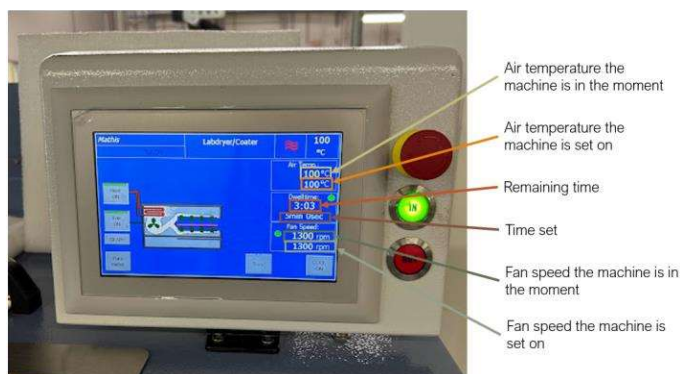


Figure 38: Display that shows the drying settings of one of the coating applications, and explanation of the values that appear on the display.



Figure 39: Application of a coating formulation. (1) Formulation poured on the substrate; (2) spreading.

3.3.9. Topcoat

In some occasions it can be necessary to add an additive to the coating surface. An example of these additives is the topcoat, which was the one that was applied in some cases in this project.

There are many types of topcoats available. The one that was mostly used in these project tests was a bio-based one.

This topcoat is ready to apply, meaning, it is not necessary to previously prepare it, or combine it with another agent for it to be ready to apply as it happens in some products. This topcoat is applied as if it was a formulation paste and it is also dried after.

3.3.10. Curing

The curing process is the process that fixes the coating to the fabric. It is similar to the drying process although the temperatures used are higher. It is done in the same machine as the drying process, and it is carried out after all the layers required were applied.

For this dissertation's purpose the parameters used for curing the experiments were 150°C of air temperature, for 5 minutes and the fan speed varied between 1300 and 2300 rpm. Figure 40 shows an example of the display during one of the curing processes done.



Figure 40: Display that shows the curing settings of a coating applications.

3.3.11. Calendering and Embossing

The final processes used in the material experiments done were the calendering and embossing. These processes were carried out in a machine available at CITEVE as the one presented in figure 41, which is a Macpi 553.37-9124 FLAT BONDING PRESS. This machine applies pressure combined with heat on the samples and alters its surface. In the embossing case, some proper papers with effects and textures were used to give the surface some texture or effect. It was used more than one type of paper in the embossing experiments, but the most used was the SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R, which in some way enhances the calender's work and makes the coating surface smoother.



Figure 41: Machine's model used to make the calendering and embossing processes.

3.4. New Coating Materials Development

In the next table an overview of the tests that were performed to reach the developed coated materials is presented. The beginning points for these experiments were based on the know-how and previous experiments done by CITEVE in other similar projects. The main goal to the final surface results were for it to look and feel like leather, so the characteristics that were tried to achieve throughout these experiments were: soft and smooth touch feeling, thickness of the material similar to leather's, so it would also "behave" like it, highly pigmented colour, and a resistant surface to abrasion and wear. It was also an objective to incorporate as much waste as possible in each material. Figure 42 shows images of all the resulting coated textiles from each experiment.

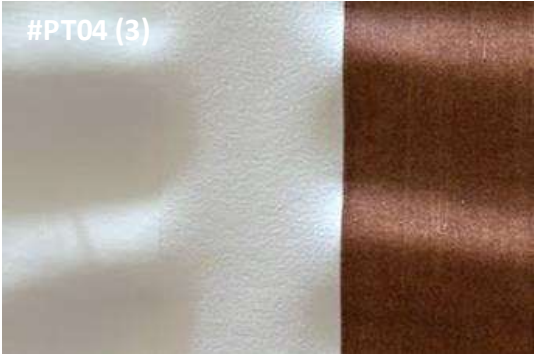
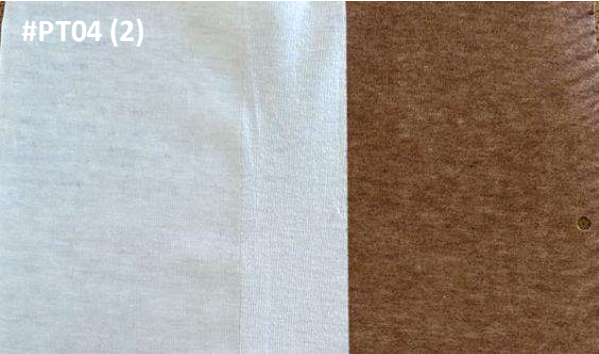
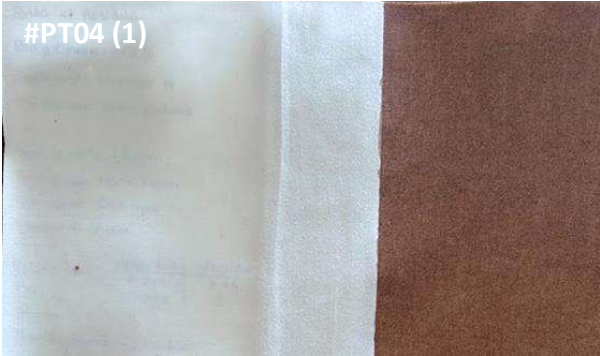
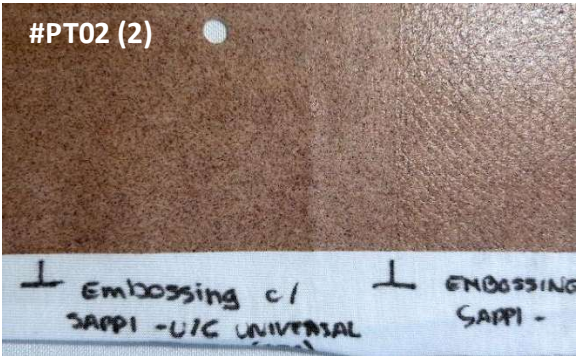
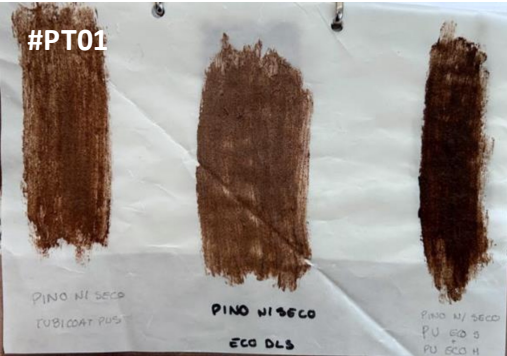
The experiments that will be more detailed are the ones that were considered the best and final results of the designed coated materials. Those are #PT12 for the material designed with pine bark and #PT14.2 is the experiment that was considered the final and best result using the pine sawdust waste.

EXPERIMENT NUMBER	OBJECTIVES	TEXTILE SUBSTRATE	PINE WOOD WASTE TYPE	% OF WASTE	CONCLUSIONS	NOTES
#PT01	Test different solutions to see which one seemed more interesting to begin with.	Recycled polyester + cotton woven fabric (thick) & Plasticised paper	Bark	11		
#PT02	Tests with POLYMER 2.	Recycled polyester + cotton woven fabric (thick) & Knitted fabric 100% cotton	Bark	5	Rough texture due to type of polymer; stiff surface; doesn't crack. More malleable; didn't transfer; didn't crack.	Woven Fabric Knitted Fabric
#PT03	Tests using POLYMER 2 without crosslinker + control paste to compare texture.	Recycled polyester + cotton woven fabric (thin) & Knitted fabric 100% cotton	Bark	5	Very hard and stiff surface. Very hard and stiff surface.	Woven Fabric Knitted Fabric
#PT04	Tests with POLYMER 4	Recycled polyester + cotton woven fabric (thin) & Elastic knitted fabric & Knitted fabric 100% cotton	Bark	5	Overall more promising than the ones in #PT03.	
#PT04.1	Tests with POLYMER 4 with incorporation of crosslinker to compare with #PT04.	Elastic knitted fabric	Bark	5	Not much different texture and surface than the paste in #PT04.	
#PT05	Tests with POLYMER 4 with incorporation of emulsifier applied in 2 different fabrics.	Recycled polyester + cotton woven fabric (thick) & Elastic knitted fabric	Bark	5	The results in these samples were unsatisfactory. It wasn't determined the cause for such bad results.	
#PT05.1	Tests with POLYMER 4 with incorporation of emulsifier and crosslinker applied in 2 different fabrics to compare with #PT05.	Recycled polyester + cotton woven fabric (thick) & Elastic knitted fabric	Bark	5	The results in these samples were unsatisfactory. It wasn't determined the cause for such bad results.	
#PT05.2	Tests with POLYMER 4 with incorporation of crosslinker, no emulsifier, using pine bark waste previously dried. Applied in 2 different fabrics to compare with #PT05 and #PT05.1.	Recycled polyester + cotton woven fabric (thick)	Bark	5	The results in these samples were unsatisfactory. It wasn't determined the cause for such bad results.	

#PT06	Test similar formulations to the ones that went wrong (#PT05; #PT05.1; #PT05.2) to try to understand what went wrong. + layer thickness experiments.	Recycled polyester + cotton woven fabric (thick)	Bark	5	Both samples PT06(1) and PT06(1.1) show a glossier surface when compared to the results in #PT06.1.	
#PT06.1	Test similar formulations to the ones that went wrong (#PT05; #PT05.1; #PT05.2) to try to understand what went wrong. + layer thickness experiments.	Recycled polyester + cotton woven fabric (thick)	Bark	5	Both samples #PT06.1(2) and #PT06.1(2.1) show a less glossy surface when compared to the results in #PT06.	
#PT07	Test an increase of waste % using the same products in #PT06 + compare results with the tests #PT08.1 and #PT08.2 made alongside.	Recycled polyester woven fabric	Bark	7	The surface is some amount rough, but it seems a more even surface when compared with the results in #PT06.	
#PT08.1 & #PT08.2	Test the use of waste ball milled to perceive the behaviour of the waste when ball milled + to try the 5% and 7% of waste in the mixtures.	Recycled polyester woven fabric	Bark	5	Bad results. Lumps and "strings" in the coating surface.	#PT08.1
				7	Bad results. Lumps and "strings" in the coating surface.	#PT08.2
#PT09.1 & #PT09.2	To test the formulation that uses 7% of pine bark waste with different layers amount and thickness.	Recycled polyester woven fabric	Bark	7	Good surface texture. Conclusion that viscosity values should be between 5-10 Pa.s.	
#PT10.1 & #PT10.2	To test different types of thickeners.	Recycled polyester woven fabric	Bark	7	Result with Thickener 1, is a smoother surface than with Thickener 2.	
#PT11.1 & #PT11.2	Changes in crosslinker type and amount + layers amount ad thickness testing.	Recycled polyester woven fabric	Bark	7	Less "strings". Crosslinker combined with the layers thickness and arrangement can be the answer.	
#PT12	Layer amount and thickness testing. Decrease of the crosslinker amount to 0.3%.	Recycled polyester woven fabric	Bark	7	Much more even and homogeneous surface.	
#PT13	To test the use of pine sawdust waste.	Recycled polyester woven fabric	Sawdust	7	No lumps or "strings". It presents an even and homogeneous surface. Very light-coloured surface.	
#PT14.1 & #PT14.2	Test the use of 10% amount of waste+ test layers amount and thickness.	Recycled polyester woven fabric	Sawdust	10	Some amount less smooth than the sample from #PT13. Darker surfaces.	
#PT15	Test obtaining darker shade surface by using different amount and thickness of layers.	Recycled polyester woven fabric	Sawdust	10	The shade is darker but the difference to #PT14.2 is not relevant.	

Note: In some cases, tests are together, for example "#PT08.1 & #PT08.2", while, in other cases they are separated as it is the case with #PT06 and #PT06.1. The reason for it to be differentiated is that, when they are presented as separate experiments, it means that there were changes in the formulation, although, does changes usually are small like, adding an additive, as so, they are considered a variation and that is why they are presented as a new experiment but are linked to another. In the cases where they are presented together, the difference lies on the layers' thicknesses and arrangement varying between them, or percentages of the same formulation components also.

Design of Bio Based Coated Textiles with Incorporation of Pine Wood Wastes



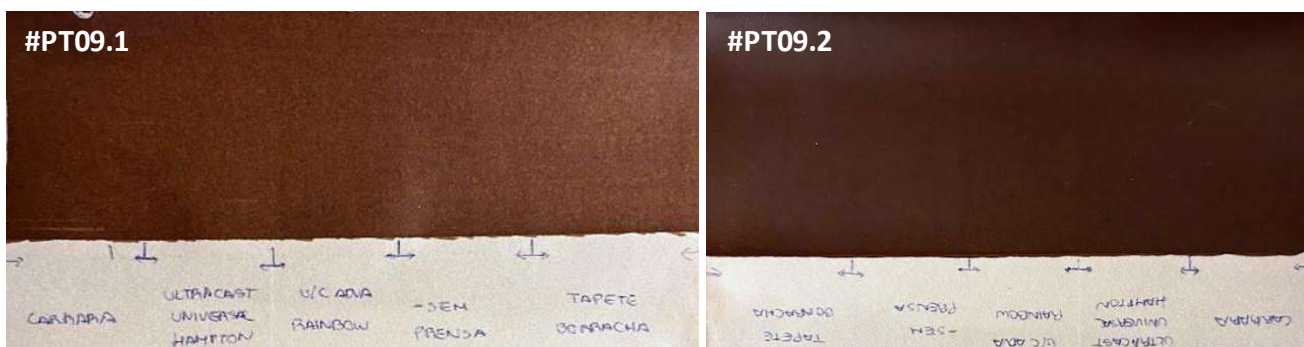
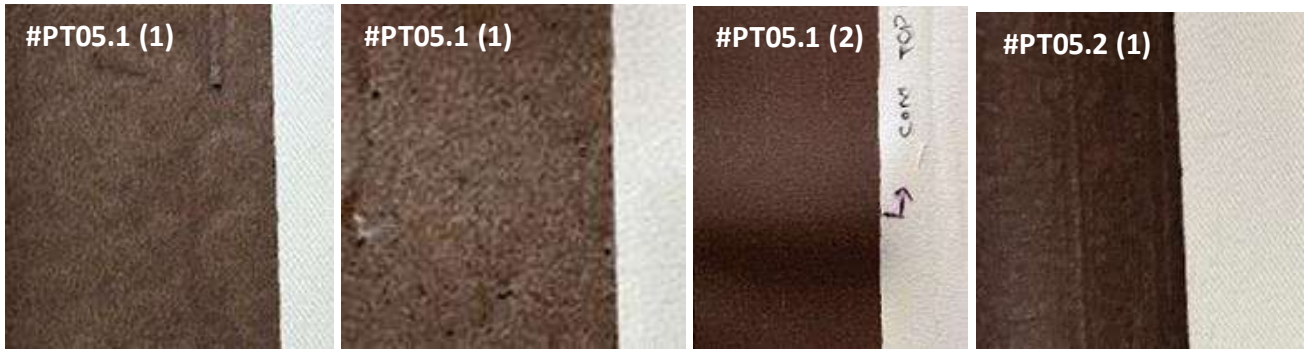
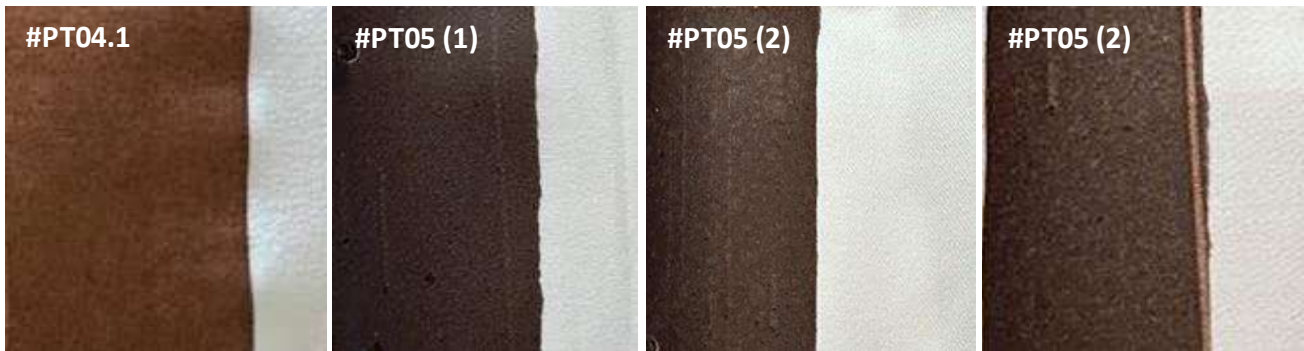




Figure 42: Images of all the results obtained in the coating experiments.

EXPERIMENT #PT12

OBJECTIVE: Number of layers and thickness testing. Decrease of the crosslinker amount to 0.3%.

DATE: 16/05/2023

TEXTILE:

TEXTILE SUBSTRATE	Recycled polyester woven fabric
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COATING FORMULATION: 300g

	DESIGNATION	AMOUNT (g)	%
POLYMER	POLYMER 4	278.1	92.7
WASTE	Pine Bark (previously dried)	21	7
ADDITIVES:Thickener	THICKENER 1	q.b	-
Crosslinker	CROSSLINKER 1	0.9	0.3
Topcoat	TOPCOAT 2	-	-

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
Scale	PG 5002 Deltarange
Mixer	Techmatic S2
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Strainer	-
Calender Machine	Macpi 553.37-9124 FLAT BONDING PRESS
Calender Papers	SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	-
DRYING	100 °C/ 5 min./ 1300 rpm*	Each layer dried separately
CURING	150 °C/ 5 min./ 1300 rpm*	After all layers applied
LAYERS	2x 0.1mm	-
CALENDERING	140 °C/ 30 seg./ 3 Bar	

*Fan Speed

OTHER INFO:

	VALUES	NOTES
VISCOSITY	7.96 Pa.s (7960 cps)	100 rpm Torque: 79.6% s06*
PH	Not measured	-

* S means Spindle and the number is the identifier of the spindle.

NOTES:

- Order of mixing was polymer, then crosslinker, then residue was added slowly, not all at the same time, while the paste was mixing continuously, then thickener was added drop by drop until visually it was less liquid, sticking to the spoon.
- The crosslinker was decreased by 0.05% to its recommended amount.
- The measurement of viscosity was made as soon as the paste was ready to be applied.
- This paste was strained 2 times before application to remove and avoid lumps in it.
- The formulation was applied in a woven fabric made of recycled polyester.
- The sample has 2 layers with 0.1mm each.
- The sample has been top coated and calendared with the SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R paper.

CONCLUSIONS/OBSERVATIONS/RESULTS:

- This test was made to try and increase the height of the layers applied to the substrate. It is believed that the fact that the first layer was being applied with 0.0mm was one of the causes of the “stringy” appearance on the coating surface. So, the test was done to experiment applying layers with 0.1mm.
- The result shows, in fact, a much more homogeneous even surface.
- This is considered to be the last test with the pine bark waste, regarding this dissertation. This doesn’t mean that this coating paste is completely ready to be industrialised. It is believed that it is very promising and can, in further studies, be improved to a more stable and homogeneous coating paste.

Figure 43 shows an image of the sample resulting from #PT12. which is the considered the developed coated material using pine bark waste.

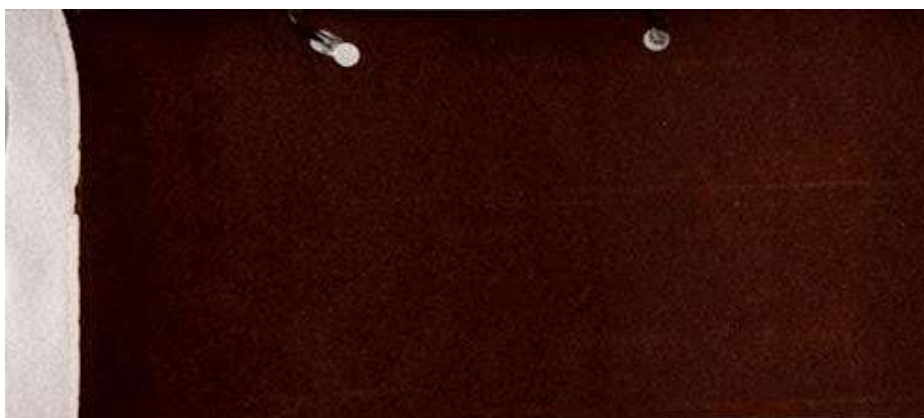


Figure 43: Sample #PT12.

EXPERIMENT #PT14.1 and #PT14.2

OBJECTIVES: Test the use of 10% amount of waste in the formulation and layers amount and thickness.

DATE: 22/05/2023

TEXTILE:

TEXTILE SUBSTRATE	Recycled polyester woven fabric
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COATING FORMULATION: 100g

	DESIGNATION	AMOUNT (g)	%
POLYMER	POLYMER 4	89.7	89.7
WASTE	Pine sawdust	10	10
ADDITIVES: Thickener	THICKENER 1	q.b	-
Crosslinker	CROSSLINKER 1	0.3	0.3
Topcoat	TOPCOAT 2	-	-

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
Scale	PG 5002 Deltarange
Mixer	Techmatic S2
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Calender Machine	Macpi 553.37-9124 FLAT BONDING PRESS
Calender Papers	SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	-
DRYING	100 °C/ 5 min./ 1300 rpm*	Each layer dried separately
CURING	150 °C/ 5 min./ 1300 rpm*	After all layers applied
LAYERS	3x 0.0mm 3x 0.1mm	14.1 14.2
CALENDERING	140 °C/ 30 seg./ 3 Bar	

*Fan Speed

OTHER INFO:

	VALUES	NOTES
VISCOSITY	3.93 Pa.s (3930 cps)	100 rpm Torque: 39.3% s06*

* S means Spindle and the number is the identifier of the spindle.

NOTES:

- Order of mixing was polymer, then crosslinker, then residue was added slowly, not all at the same time, while the paste was mixing continuously, then thickener was added drop by drop until visually it was less liquid, sticking to the spoon.
- The measurement of viscosity was made as soon as the paste was ready to be applied.
- The paste was applied in a woven fabric made of recycled polyester.
- This test presents 2 different samples, 14.1 and 14.2, both using the same paste. #PT14.1 has 3 layers with 0.0mm each and #PT14.2 has 3 layers with 0.1mm each.
- In part of the samples, a topcoat was applied to test how it looked and felt in this coating as well as part was also embossed using the paper SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R.

CONCLUSIONS/OBSERVATIONS/RESULTS:

- This test was done to try increasing the % of waste in the paste from 7% (the amount used in the first test with the sawdust) to 10%. The increase did not create problems, in fact it delivers a more homogeneous surface.
- The surface is somewhat less smooth than the sample from #PT13.
- #PT14.1 has the same number and layers and height as #PT13. There is a slight change in colour although it is not significant. Which means the colour intensity doesn't come from the increase of percentage of waste in the formulation.
- #PT14.2 has thicker layers, and the results are visible. It acquired a darker shade, and the surface looks more homogeneous. Also, the touch feels smoother.
- A topcoat was applied in a part of both samples: in both the surface becomes smoother and matter (less glossy) as well as less sticky. In #PT14.1 it is still possible to feel some rough surface but in #PT14.2 it acquires a very smooth surface.
- Both samples were embossed, in the part where topcoat was applied as well as in the part that doesn't have topcoat. In the part that was topcoated the embossing made the surface softer for both samples. The same happened in the part that didn't have topcoat but the difference between this part, embossed or not embossed, it's not as prominent as the difference in the part that has topcoat.

Figure 44 shows an image of the sample resulting from #PT14.2 which is the considered the developed coated material using pine sawdust waste.



Figure 44: Sample #PT14.2.

3.5. Conclusions on the material development

The considered final experimental materials, resulting from this dissertation' study are, as said before, the ones achieved in #PT12 in the case of the material that was developed with the Portuguese derived pine bark waste and in the case of the material designed with the Chilean pine sawdust it was #PT14.2

These results present intense and pleasing colours and the surfaces are soft and smooth, especially the result with pine bark waste, due to the combination of topcoat and calendering that were applied in the coating surfaces.

These materials are considered experimental, more sustainable alternatives to leather. As such, it is believed that they have similar characteristics to leather and can eventually be used for similar purposes.

4.DESIGNED MATERIALS FORMULATIONS AND SURFACE TESTS

4.1. Introduction

To have some preliminary information on the characteristics and resistance of the material being designed, several tests were carried out along the process in order to achieve different types of information. The coating formulations undergo three types of tests: TGA; DSC and FTIR. these allowed to compare within each other and to compare with the same formulation without waste incorporated in it.

The resulting materials surfaces were also tested for colour fastness to washing, colour fastness to direct sunlight exposure, resistance to abrasion Martindale surface test (ISO 5470-2), colour fastness to rubbing test (ISO 105-X12) colour fastness to artificial light (ISO 105-B02:2014) and sewability.

The tests performed will be presented next in this chapter as well as the results achieved.

4.2. Thermogravimetric Analysis (TGA)

According to (Sarfraz, Raza, Mirzaeian, Abbas, & Raza, 2022):

'Thermogravimetric analysis or thermal gravimetric analysis (TGA) is a method of thermal analysis in which changes in physical and chemical properties of materials are being observed as a result of increase in temperature.' (Sarfraz, Raza, Mirzaeian, Abbas, & Raza, 2022).

To make a TGA test, a small sample (between 5-20 mg) is heated in a controlled atmosphere and as it is heated, the changes in its weight are measured. This test provides insights into the composition and thermal stability of the material by determining the temperature at which the material loses or gains mass.

This method has been used to determine the biochemical composition of many materials such as beechwood, switchgrass, corn stover, ... (Brown, Gallagher, & Hwang, 1998).

This test was performed to the formulations by Leonardo Santana at FEUP.

4.2.1. TGA Results

The TGA was made to the formulation with pine bark waste, the one with pine sawdust waste and also to a “control” formulation that contained the same ingredients to the other two formulations mentioned except the waste.

In the thermogravimetric curve, shown in figure 45, resulting from the analysis of the “control” formulation (without wastes), it is visible a plateau that can indicate the release of some solvent or water as there is a decrease of around 5% on the formulation's weight.

As for the TGA curve correspondent to the formulation with pine bark waste, and the formulation with pine sawdust waste, both visible in figure 45, this level does not exist. This can be promoted due to some type of reaction of the waste with the polymer that integrates the formulation.

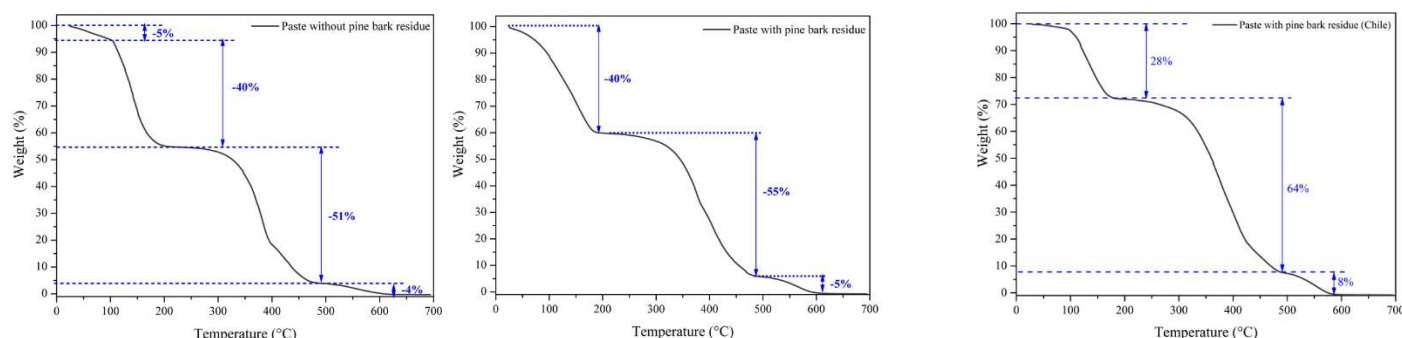


Figure 45: Graphics with the results of the TGA tests.

Also, in the main mass loss plateaus the beginning degradation temperature, tends to decrease slightly in the formulations with wastes when compared to the one without waste.

Regarding bigger mass losses, taking the example of 50% weight loss, represented in the graphics in figure 46 it is also necessary a higher temperature to reach this percentage of mass loss in the formulation with pine bark waste (345°C) when compared to the “control” formulation (326°C). As for the formulation with sawdust waste it even has a better behaviour compared to the two previously mentioned as it only reaches 50% mass loss at 360°C.

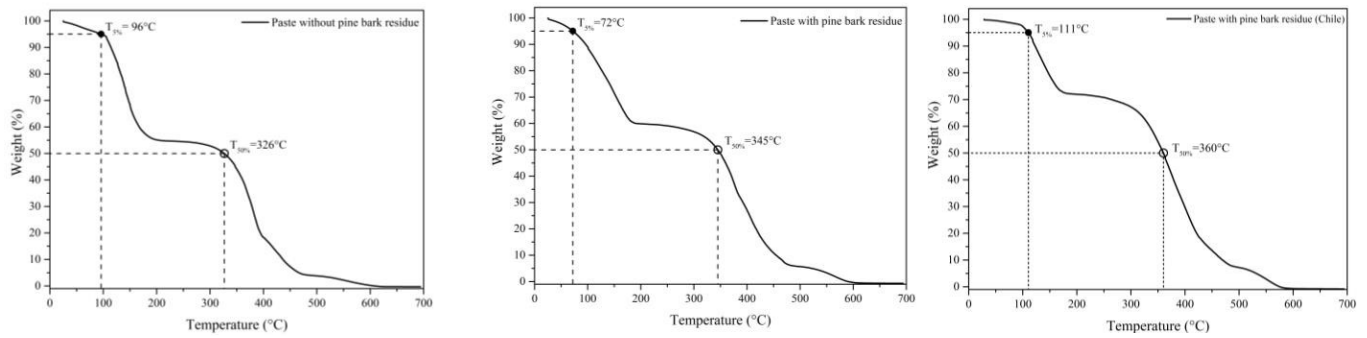


Figure 46: Graphics with the results of the TGA tests. Indication of the temperature at which there is a 50% mass loss in each formulation.

Overall, it was concluded that the insertion of the wastes makes this coating formulations slightly more resistant to high temperatures than the same formulation without the waste, being the most thermally stable the formulation with pine sawdust waste.

4.3. Differential Scanning Calorimetry (DSC)

DSC is a technique used to get information about a material in thermal properties such as temperature, melting point, specific heat capacity, cure process, oxidation behaviour, thermal stability, ...

In this technique, the material's sample is placed in the DSC machine in which heat flows into or out of the sample and time or temperature are measured (Ahmadi Khoshooei, Fazlollahi, & Maham, 2019).

This test was performed to the formulations by Leonardo Santana at FEUP.

4.3.1. DSC Results

The DSC was only possible to performed on the formulation without any waste, the “control” and the formulation with pine bark waste.

The graphics that represent the results of the DSC tests are presented in figure 47.

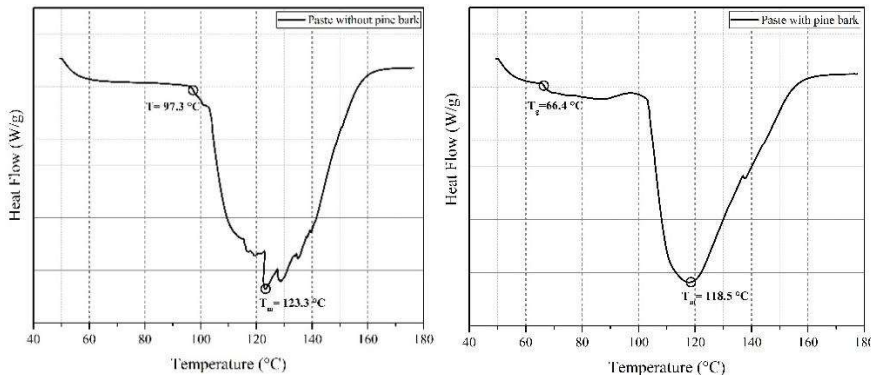


Figure 47: Graphics with the results of the DSC tests.

It was observed that the formulation without waste presents higher glass transition (T_g) and melting temperatures (T_m), $T_g = 97.3^\circ\text{C}$ and $T_m = 123.3^\circ\text{C}$, than the formulation with pine bark waste, $T_g = 66.4^\circ\text{C}$ and $T_m = 118.5^\circ\text{C}$. The investigator that performed this analysis believes that the T_g of the formulation without waste is 97.3°C although he was not certain.

It was also observed that the temperature transitions are more evident in the formulation with pine bark waste.

With this it was possible to conclude that the incorporation of pine waste reduces the glass transition and melting temperatures. This is considered to be beneficial because it has an impact on the amount of energy required for the production.

4.4. Fourier Transform Infrared Spectroscopy (FTIR)

According to (Berna, 2017),

'FTIR is an instrumental technique used to identify the functional groups present in organic and inorganic compounds by measuring their absorption of infrared radiation over a range of wavelengths' (Berna, 2017).

To do so, FTIR starts by collecting an "interferogram of a sample signal using an interferometer, and then it performs a Fourier transform (a mathematical algorithm) on the interferogram to obtain the infrared spectrum." After this what the FTIR does is digitise what it collected and shows the FTIR spectrum. To do this test, only tens of micrograms of the material's sample are needed (Berna, 2017).

This test was performed to the formulations by Leonardo Santana at FEUP.

4.4.1. FTIR Results

The FTIR was performed in all three formulations mentioned in the previous tests, the “control” formulation, without waste; the formulation with pine bark waste; the one with pine sawdust waste and in this test’s case, it was also performed in the base polymer that is present in all the formulations, POLYMER 4.

The following graphic in figure 48 shows the results of each FTIR in each formulation.

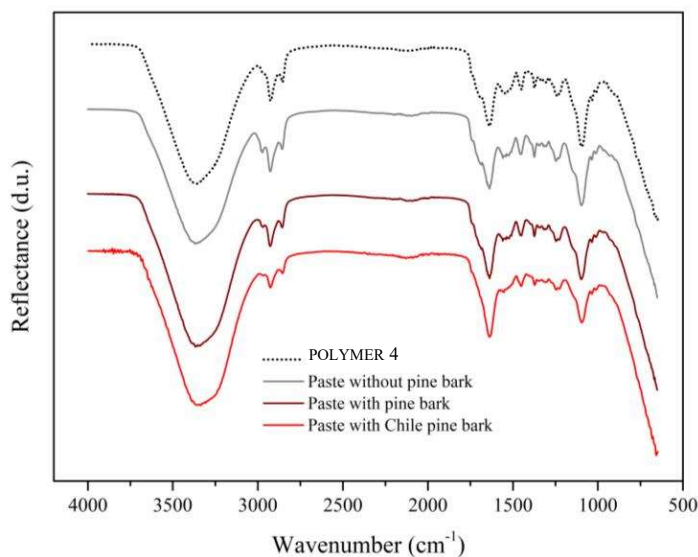


Figure 48: Graphic with the results of the FTIR tests (adapted).

As it is visible in the above graphic, the waves correspondent to the formulations, with and without wastes, are all very similar to each other. As so, it was concluded that there isn't any significant difference in the molecular structure of these formulations that should be considered or mentioned.

Although, when comparing the waves that represent the FTIR from the formulation without waste and the one that represents the POLYMER 4, it is visible small, but more visible changes. These changes are believed to be related to the presence of the additives that the formulations have that the polymer alone doesn't have.

4.5. Colour fastness to washing and colour fastness to direct sunlight exposure

Both materials developed were tested for sunlight exposure and washing cycles exposure. A sample of each material was exposed in certain conditions that will be described next.

Colour fastness to direct sunlight exposure test conditions

A sample of each designed material was tested to determine its resistance to sunlight. This test involved putting the sample in a place with direct sunlight progressively covering parts of the sample with a black cardboard as the days went by, so that the covered parts cease to receive direct sunlight exposure. In this case the samples were sectioned in 6 parts, one that wouldn't be exposed, one that would be exposed for 1 day, another for 2 days, another part for 7 days, 14 days and the last part, 21 days.

In figure 49 it is shown the samples of the materials developed that were tested for sunlight resistance.

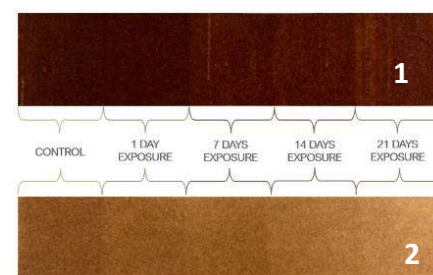


Figure 50: Samples that were exposed to direct sunlight. (1) Sample with pine bark waste; (2) Sample with pine sawdust waste.

Colour fastness to washing test conditions

A sample of each developed material was tested to assess their colour fastness resistance to washing.

To do so, the samples were divided in 5 stripes, one that wouldn't be washed, one was washed once, another was washed 5 times, another 10 times and the last 15 times.

All of the washing cycles were made in the same conditions, 40°C; for 30 minutes in a Samsung washing machine, model WF90F5E2W2W Eco Bubble 9 Kg, as the one shown in figure 50 using the commercial liquid clothes washing detergent from Persil as also shown in figure 50.

In figure 51 it is shown the samples of the material with the pine bark waste, and the samples with the pine sawdust waste.



Figure 51: Machine and product used in the washing exposure tests. (1) washing machine model used; (2) Washing detergent used.



Figure 49: (1) Samples of the material designed with pine bark waste that were tested for colour fastness to washing; (2) Samples of the material designed with pine sawdust waste that were tested for colour fastness to washing.



Figure 52: Datacolor® Spectraflash® SF450® - the machine that “reads” the samples.

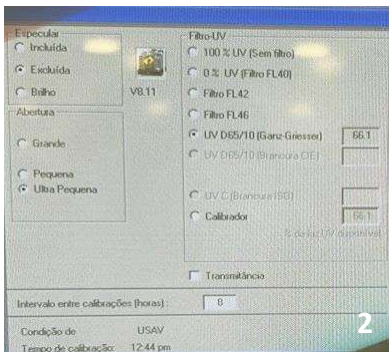


Figure 53: Some of the tools and settings used to calibrate Datacolor® Spectraflash® SF450®. (1) Aperture used for the calibration and tests. (2) Settings for the calibration.

4.5.1. Colour analysis of the sunlight exposure and washing cycles tests

After the exposure was complete, the samples were analysed in a type of spectrophotometer. This tool analyses the colour of the sample, giving accurate colorimetric data with the opportunity to compare it to a pattern sample which is what was performed in this test. The machine used was the Datacolor® Spectraflash® SF450® in combination with the reading software Datacolor® Colortools® (Figure 52).

The first step to perform this analysis was to calibrate the machine. This procedure starts by selecting the calibration option in the software and setting the parameters correctly and according to the aperture that is in the machine. In this case the aperture used was the “Ultra Small” (“Ultra pequena”) which is an aperture of 6.6 mm that can be seen in figure 53 and the other settings can also be checked in figure 53. Then, it is necessary to follow the instructions on the screen that indicate to put standard tools in the machine for it to “read” those, one black and one white. These standards are like the ones in figure 54.



Figure 54: Patterns to calibrate the machine Datacolor® Spectraflash® SF450®. (1) and (2) different views of the black standard for the calibration; (3) and (4) different views of the white standard for the calibration

After these readings the machine should be ready to analyse the samples.

Initially a pattern must be set, which is why there is a sample in each test that wasn't exposed. This piece is what the other samples will be compared to.

The software provides quantitative results for CIELAB colour space, K/S values and the ΔE which is the only quantitative value that is given by comparing the colour of the pattern and the samples being studied.

CIELAB (also known as Lab or Lab*) is a colour space used in colour science and digital imaging. It was developed by the International Commission on Illumination (CIE) and it is known to be the most accurate colour space, mainly used in the plastics, automotive and textile industry. It consists of three components: L^* that represents the lightness component of the colour and ranges from 0 to 100, 0 representing black, total darkness and 100 white. The a^* component represents the red-green axis and can show values from -128 to +127. Negative values represent a greener prominence while positive values indicate redness. The b^* component represents the axis of yellow-blue and the values also go between -128 to +127. In this case negative values indicate blueness and positive values indicate yellowness (Hermans, 2023).

The K/S value is used in the textile industry to characterise fabrics' colour strength or colour intensity. The higher the k/s value is, the more intense or saturated the colour of the fabric surface is while lower values indicate less intense or saturated values (Cui & Shao, 2021).

As for ΔE , as said before it is the only value that is given by comparing to a given pattern. It is the difference between the displayed colour and the original colour pattern. The values can range from 0 to 100 and the lower the ΔE value is, the closer the colours are, while higher values indicate a significant difference between the colours (ViewSonic, 2023).

As for qualitative results these are all given by comparing the sample being studied to the pattern set. The software indicates if the studied sample is darker or brighter than the set pattern; if it is more or less saturated than the set pattern and if it is closer to one of the four colours used in the CIELAB colour space, blue, green, yellow and red than the set pattern.

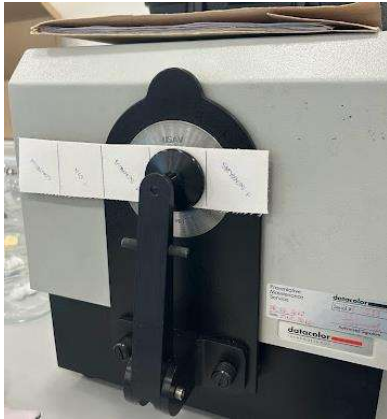


Figure 55: Positions in which the samples were read.

To set this pattern, one should name it in the software and make a normal reading of the sample. There are two normal ways to read samples. One that only reads the sample once and another that reads it 3 times (or more but 3 is the most common) and makes the average of the 3 readings. The method used was this last one. In this method it is also common to move the sample to different positions or different spots in between readings to make it more accurate. In this case, the samples' readings occurred with them in a horizontal, oblique, and vertical position as shown in the figure 55.

All of the samples resulting from the washing tests as well as the sunlight exposure tests were analysed through this process. The following tables 3 and 4 show the results of this analysis are provided.

4.5.2. Results and conclusions of the sunlight exposure tests

4.5.2.1. Material designed with pine bark waste

As it is visible to the “naked eye” in figure 49 (1) presented before, there are clear changes in the material's surface shade that begin to be visible from the part that was exposed for one day to the part that was exposed for seven days, where the biggest difference between the shade in the “Control” area, to the shade in the area of the sample that was exposed for twenty-one days.

This is what is visible just by looking at the sample. In table 3 the results of the colour analyses made to these samples in the Datascolor® Spectraflash® SF450® are presented:

Table 3: Results of the analysis made in the Datacolor® Spectraflash® SF450® to the designed material with pine bark waste samples that were exposed to sunlight.

Sample designation	L*	a*	b*	ΔE	K/S	Qualitative results
Light Control Pine Bark	32.17	15.20	14.66	-	12.342	-
Light 1 PB (1 day exposure)	31.04	16.03	14.31	1.45	12.906	darker, more saturated, more red
Light 1s PB (7 days exposure)	33.83	17.93	17.85	4.52	12.464	lighter, more saturated, more yellow
Light 2s PB (14 days exposure)	36.25	18.71	20.21	7.73	11.517	lighter, more saturated, more yellow
Light 3s PB (21 days exposure)	38.07	19.20	22.32	10.46	11.828	lighter, more saturated, more yellow

With these results it is possible to conclude that there is not much difference between the Sample that wasn't exposed to the one that was exposed for one day. However, from the sample that was exposed for seven days to the one that was exposed for twenty-one days, the surface colour gets lighter and closer to a yellower shade as it is possible to see by looking at the "b*" column: the values increase 2 points in between samples. The shade gets less close to the "original" colour as many days it is exposed to direct sunlight as it is possible to assess from the ΔE column.

This means that this material suffers surface shade changes when exposed to direct sunlight.

4.5.2.2. Material designed with pine sawdust waste

In this material's case, as it is visible to the "naked eye" in figure 49 (2), there is also a change in the material's surface shade that

begins to be visible from the part that was exposed for one day to the part that was exposed for seven days, although, it is less noticeable than the changes in the material designed with pine bark waste. Also, in this case the biggest difference in the surface shade is between the “Control” and the sample that was exposed for twenty-one days.

As for more accurate results on the differences between the exposed samples and the “Control” sample, table 4 shows the results of the colour analyses made to these samples in the Datacolor® Spectraflash® SF450®:

Table 4: Results of the analysis made in the Datacolor® Spectraflash® SF450® to the designed material with pine sawdust waste samples that were exposed to sunlight.

Sample designation	L*	a*	b*	ΔE	K/S	Qualitative results
Light Control Sawdust (Sd)	60.35	13.00	29.35	-	6.1796	-
Light 1 Sd (1 day exposure)	63.38	12.68	30.58	3.29	5.2490	lighter, more saturated, more yellow
Light 1s Sd (7 days exposure)	63.56	12.72	32.63	4.60	5.7831	lighter, more saturated, more yellow
Light 2s Sd (14 days exposure)	66.05	11.58	34.04	7.52	5.6437	lighter, more saturated, more yellow
Light 3s Sd (21 days exposure)	68.47	10.66	34.81	10.07	5.3321	lighter, more saturated, more yellow

With these results it is possible to conclude that, contrary to what is visible to the naked eye, there is a greater difference between the “Control” sample and the sample that was exposed for one day. It is visible in the ΔE column that there is a difference of three points in between these two shades.

In this case, contrary to what happens in the material designed with pine bark waste, the smallest difference between shades occurs between the sample that was exposed for one day and the sample that was exposed for 7 days, while in the pine bark waste material the littlest difference in shades is between the “Control” sample shade and the sample shade that was exposed for one day.

As happens in the material that was designed with pine bark waste, this sample also gets lighter as it gets more exposure to sunlight as well as acquiring a yellower shade as it is possible to see by looking at the “b*” column: the values increase in between samples, however, the difference is less prominent than the difference between shades in the material shown in table 4. The shade gets less close to the “original” colour as many days it is exposed to direct sunlight as it is possible to assess from the ΔE column.

It is possible to conclude that this material suffers surface shade changes when exposed to direct sunlight like the previous material did, however, they suffer changes differently and on different periods of the exposure.

4.5.3. Results and conclusions of the washing exposure tests

4.5.3.1. Material designed with pine bark waste

Contrary to what happens with the samples that were tested for sunlight exposure in these samples it is not visible to the “naked eye” changes in the coating surface of the samples tested. However, there were indeed some surface changes detected in the analysis made in Datacolor® Spectraflash® SF450®.

The results of this analyses are presented in table 5 next:

Table 5: Results of the analysis made in the Datacolor® Spectraflash® SF450® to the designed material with pine bark waste samples that were exposed to washing cycles.

Sample designation	L*	a*	b*	ΔE	KS	Qualitative results
Control Pine Bark (PB)	31.50	14.83	14.66	-	12.989	-
Washed 1x PB	31.32	14.80	14.38	0.34	12.660	darker; less saturated, more red
Washed 2x PB	30.57	15.31	13.80	1.35	13.131	darker; less saturated, more red
Washed 5x PB	29.30	15.26	13.65	2.46	14.631	darker; less saturated, more red
Washed 10x PB	29.28	15.78	13.42	2.71	14.231	darker; less saturated, more red
Washed 15x PB	28.50	14.36	10.79	4.92	13.303	darker; less saturated, more red

In these results it is possible to conclude that overall, there are some changes to this coating surface. It gets a less saturated colour from getting washed however these changes are not as significant as it is visible in the ΔE column. The biggest difference of surface colour occurred between the sample that was washed 10 times to the one that was washed 15 times. While until the sample that was washed 10 times the difference between the “Control” sample and the washed samples surface colour was between 0.34 and 2.71, meaning a difference of 0,25 to 1,11 points in between washed samples, in the sample that was washed 15 times it spikes to a difference to the “Control” sample of 4.92 points which translates to a difference of 2,21 points between the sample that was washed 10 times and the one washed 15 times. Either way, when compared to the results obtained in the sunlight exposure tests the difference between surface shades is much less evident.

4.5.3.2. Material designed with pine sawdust waste

Much like the results presented before in the other designed material, this one also doesn't show much difference in the surface shade to the “naked eye”.

However, some changes did occur in between washings of the material designed with pine sawdust waste when taking into account the results of the analysis made with Datacolor® Spectraflash® SF450® on the washed samples.

This analysis is presented below in table 6.

Table 6: Results of the analysis made in the Datacolor® Spectraflash® SF450® to the designed material with pine sawdust waste samples that were exposed to washing cycles.

Sample designation	L*	a*	b*	ΔE	KS	Qualitative results
Control Sawdust (Sd)	57.15	13.24	28.77	-	6.7429	
Washed 1x Sd	57.33	13.03	27.37	1.43	6.2279	lighter, less saturated, more red
Washed 2x Sd	58.03	13.42	27.81	1.32	6.1475	lighter, less saturated, more red
Washed 5x Sd	57.24	13.31	27.20	1.57	6.2320	lighter, less saturated, more red
Washed 10x Sd	55.03	13.10	25.69	3.75	6.6429	darker, less saturated, more red
Washed 15x Sd	55.41	13.20	25.68	3.55	6.1161	darker, less saturated, more red

In this case the results of the analysis also show some changes to the surface shade of the samples when compared to the “Control” one. The changes are not very significant however, by looking at the ΔE column, the difference between the “Control” and the sample washed once (1.43) is greater than the difference between the same two samples in the test with the material designed with the pine bark waste (0.34).

Contrary to what happens in the tests with the material using the pine bark waste, in this case, when considering the results in the ΔE column, the biggest difference of surface colour occurred between the sample that was washed 5 times to the one that was washed 10 times. Until the sample that was washed 5 times, the difference between the “Control” sample and the washed samples surface colour was between 1.32 and 1.57 meaning a difference of 0,11 to 0.25 points in between washed samples.

In the sample that was washed 10 times it spikes to a difference to the “Control” sample of 3.75 points which translates to a difference of 2,18 points between the sample that was washed 5 times and the one washed 10 times. The sample that was washed 15 times also shows a larger difference between surface shade when compared to the “control” sample (3.55), however it presents a lower difference than the sample that was washed 10 times (3.75).

Overall, the results show that the washed samples get a less saturated shade when compared to the “Control” sample.

4.6. Martindale abrasion resistance tests (ISO 5470-2)

The Martindale Tester is a machine used to test the abrasion resistance of fabrics.

This test, used in the textile industry, is made with a rotating circular table to which is attached a piece of fabric that is repeatedly rubbed against an abrasive surface to simulate the wear and tear of day-to-day use.

This test allows one to determine the fabric’s abrasion resistance by recording the number of cycles that is required to get visible wear/tear on the fabric (Wang X., et al., 2008).

Figure 56 shows a schematic diagram of the Martindale tester.

This test was performed in the testing labs at CITEVE and delivered as a report.

The conditions used to perform this test were the following:

- Type of abrasive: Wool Fabric;
- Applied pressure: 12KPa;
- Used method: 1;
- Conditioned environment: $20\pm 2^{\circ}\text{C}$ and $65\pm 4\%$ H.R.;
- Classification in between 0 and 5 in which 0 is no alteration and 5 is complete degradation.

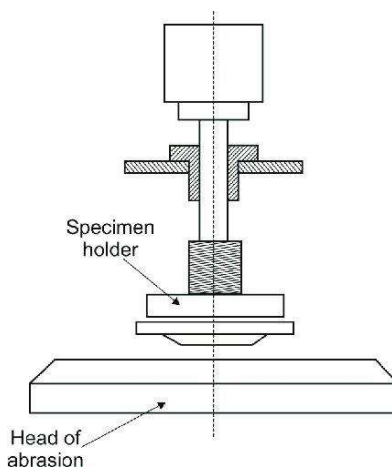


Figure 56: Schematic diagram of the Martindale Tester.

4.6.1. Results of the Martindale abrasion test

4.6.1.1. Material designed with pine bark waste

The results obtained for this test on the material developed with pine bark waste are shown in the following table 7.

In this table it is possible to see the degree of degradation between the defined number of revolutions.

Table 7: Results of the Martindale abrasion test on samples of the designed material with pine bark waste

Number of revolutions	Sample 1	Sample 2	Sample 3	Sample 4
Initial state	0	0	0	0
1600 revolutions	0	0	0	0
3200 revolutions	0	0	0	0
6400 revolutions	0	0	0	0
12800 revolutions	1	1	1	1
25600 revolutions	1	1	1	1
38400 revolutions	2	2	2	2
51200 revolutions	2	2	2	2

As it is visible in table 7, in all four samples that were tested, the material only starts showing signs of degradation from 12800 revolutions and even at 51200 revolutions it is classified as degree 2 of degradation which, being the scale between 1 and 5 indicates low degradation. As so, it is possible to conclude that the results obtained in this test are very satisfactory, showing that the material tested does indeed have a very good resistance to abrasion.

4.6.1.2. Material designed with pine sawdust waste

The results obtained for this test on the material developed with pine sawdust waste are shown in table 8. In this table it is possible to see the degree of degradation in between the defined number of revolutions.

In this material it was only possible to perform the test in two samples instead of four like shown for the material designed with pine bark waste.

Table 8: Results of the Martindale abrasion test on samples of the designed material with pine sawdust waste.

Number of revolutions	Sample 1	Sample 2
Initial state	0	0
1600 revolutions	0	0
3200 revolutions	0	0
6400 revolutions	0	0
12800 revolutions	0	0
25600 revolutions	0	0
38400 revolutions	1	1
51200 revolutions	1	1

As it is visible in table 8, the two samples that were tested, show equal results. The material only starts showing signs of degradation from 38400 revolutions and it is classified as degree 1 of degradation which, being the scale between 1 and 5 indicates very low degradation. As so, it is possible to conclude that the results obtained in this test are even better than the ones obtained in the previous shown same test performed on the material designed with pine bark waste. In this case the highest degree of degradation that is registered in the surface is 1 while in the other material it reaches number 2. The material tested has a very good resistance to abrasion and when compared to the other designed material it is considered to have a better abrasion resistance.

4.7. Colour fastness to rubbing test (ISO 105-X12)

The colour fastness to rubbing test, also known as ISO 105-X12 test is a method used to determine the resistance of the colour of textiles to rubbing or staining other materials. This test is done by subjecting the material to mechanical rubbing or friction to assess the probability of transferring colour from one material to another during normal wear.

To make this test it is used a crockmeter also known as rubbing fastness tester, which is represented in the schematic diagram in figure 57. The test is performed by rubbing a specified dry or wet white cloth against the surface of the material being tested using a specified amount of pressure.

The designed materials were tested in this method using the dry and the wet cloth procedure.

The results are reported in a scale of 1 to 5 in which 5 indicates no colour transfer or staining and 1 indicates severe colour staining or transfer (NSAI Standards, 2016).

This test was performed in the testing labs at CITEVE and delivered as a report.

The conditions used to perform this test using the dry cloth were the following:

- Used friction finger: round;
- Applied force: 9 N;
- Tested direction: web and weft or length and width;
- Conditioning conditions: 4 hours at $20 \pm 2^\circ\text{C}$ and $65 \pm 4\%$ H.R..

The conditions used to perform this test using the wet cloth were the following:

- Used friction finger: round;
- Applied force: 9 N;
- Tested direction: web and weft or length and width;
- Percentage of wetness: 95% to 100%;
- Conditioning conditions: 4 hours at $20 \pm 2^\circ\text{C}$ and $65 \pm 4\%$ H.R..

4.7.1. Results of the colour fastness to rubbing test

4.7.1.1. Material designed with pine bark waste

The result obtained for this test on the material developed with pine bark waste using the dry cloth procedure is a staining classified in the test scale as 4-5.

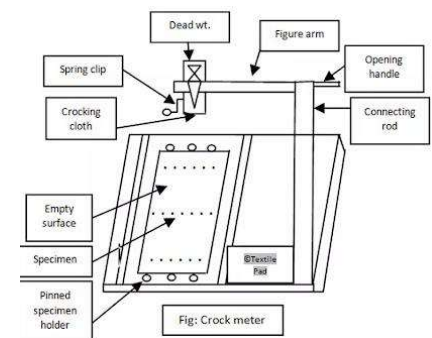


Figure 57: Schematic diagram of a crockmeter.

This means that there is no colour transfer to very slight colour transfer or staining when the material is rubbed. As for the procedure using the wet cloth the staining is also classified as a 4-5, meaning the same.

It is possible to conclude that this material has very good resistance to rubbing, and that it most likely won't transfer or stain other materials when in rubbing contact with those, which means it is possible to consider it a durable material.

4.7.1.2. Material designed with pine sawdust waste

The result obtained for this test on the material developed with sawdust waste using the dry cloth procedure presents a staining classification of 4-5. This means that there is no colour transfer to very slight colour transfer or staining when the material is rubbed. The results in the procedure using the wet cloth the staining is classified as a 4, In this case it means that this material when rubbed with the presence of a wet condition transfers or stains a very small amount.

It is possible to conclude that this material has also very good resistance to rubbing, and at least in dry conditions it most likely won't transfer or stain other materials when in rubbing contact with those, however, there is a slightly higher chance that in wet conditions it may stain or transfer some amount.

4.8. Colour fastness to artificial light: Xenon arc fading lamp test (ISO 105-B02:2014)

The objective of this test is to determine the effect on the surface colour of a textile of an artificial light source. It focuses on the resistance of textiles to fading or discoloration when exposed to light.

This test evaluates the changes in the surface colour before and after exposure to the artificial light and delivers an indication of the material's resistance to fading.

To perform this test, the sample of fabric being tested is conditioned to a specific humidity and temperature before the test begins and then it is exposed to the artificial light source. The exposure is carried out in different cycles, each cycle being determined by the amount of time the sample is exposed to light followed by a period of darkness or a new set of conditions.

The number of cycles varies between test requirements.

The results of this test are delivered using many colour measurement parameters, however, they can also be presented in a scale used to rate colour fastness based on the degree of colour change observed, in which the values go from 1 (very low colour fastness to light) to 8 (very high colour fastness to light) (BSI Standards, 2014) or it can be presented in values from 1 to 5, the same scale used to classify the results in colour fastness to rubbing test. This last one was the one used.

This test was performed in the testing labs at CITEVE and delivered as a report.

The conditions set to perform this test on the designed materials were:

- Used device: Xenon lamp air cooled;
- Used method: 3 (BS-5);
- Standard black thermometer temperature: $47 \pm 3^\circ \text{C}$;
- Humidity test: degree 5;
- Blue scale assessment.

4.8.1. Results of the colour fastness to artificial light test

4.8.1.1. Material designed with pine bark waste

The result obtained in this test on the material developed with pine bark waste classified using the rate colour fastness, was a degree of colour fastness to light of 3-4. It is considered to be good result. The results are similar to the ones obtained in the test presented in this chapter in point 4.5.2.1 where a sample of the same fabric was exposed to direct sunlight. It is possible to conclude that this material could be improved regarding its resistance to light exposure but depending on the application this degree of resistance could be acceptable.

4.8.1.2. Material designed with pine sawdust waste

The results obtained in this test on the material developed with pine sawdust waste were classified with a degree of colour fastness to light of 4-5. It is considered to a very good result.

In this case the results are also similar to the ones obtained in the test presented in this chapter in point 4.5.2.2 where a sample of the same fabric was exposed to direct sunlight. It is possible to conclude that this material could be improved regarding its resistance to light exposure.

4.9. Sewability testing

The last test to be performed in the materials designed was a sewability test. This is a very informal test, but it was done to test the ability or resistance of the materials to being sewed. As they are textile materials, eventually it would be necessary to sew them to make some type of product and so it is necessary to guarantee that the materials don't break or tear when sewed.

This test was performed on a sample of each material by sewing this sample with different types of stitchings and tensions.

The sewing machine used to perform this test was a Pfaff Quilt 1510 like the one in figure 58.

Each sample of the materials was sewed using different types of seams (see figure 59) and the settings of each type of these stitches performed are presented in table 9.



Figure 58: Sewing machine used.

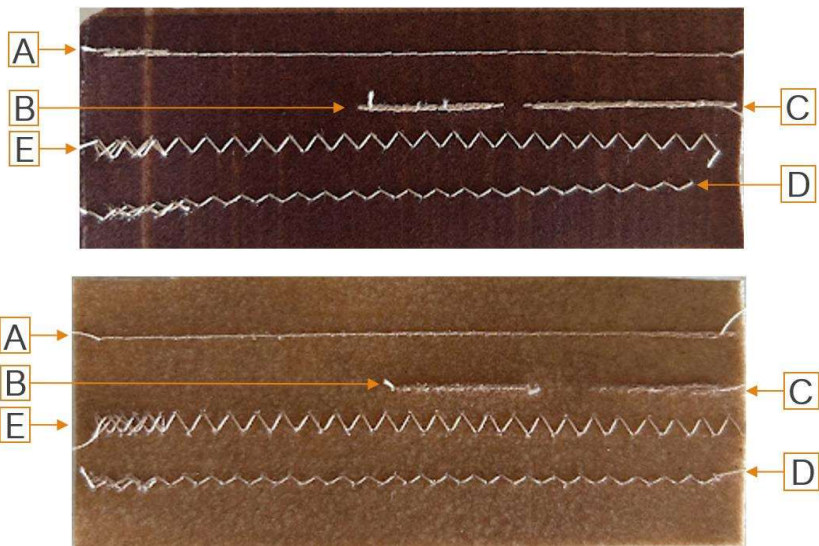


Figure 59: Samples with the testing sewings.

Table 9: Settings of the sewing machine used to perform the sewability test.

	Stitch Program	Stitch Type	Stitch Length	Stitch Width	Tension
A	E	Straight	2	-	4
B	E	Straight	Stretch	-	2
C	E	Straight	Stretch	-	4
D	E	Zig-zag	2	1	2
E	E	Zig-zag	2	3	4

4.9.1. Results of the sewability test

As it is possible to see in figure 59, none of the samples had a breakage or significant tearing resulting from the seams.

The material designed using the pine bark waste responded well to all types of seams performed on it.

The material designed with the pine sawdust waste shows a good response also to most of the stitches except stitches B and C. In the areas where these were seamed, it is visible some breakage of the coating surface, more perceptible in seam C in this sample.

This, however, is considered not to be a problem since the material is not stretchy, and most likely won't be sewed using similar types of seams.

5.SHOWCASING OBJECT DEVELOPMENT



Figure 60: Variety of pinecones

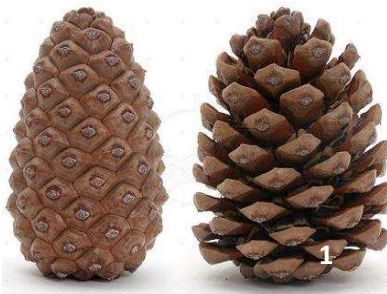


Figure 61: (1) Pinecone in closed state and open state;(2) Close up on the petals of a pinecone

5.1. Object's inspiration

The inspiration for the development of the example object was the tree from where the waste comes from, the Pine tree. In fact, the inspiration comes from this type of tree's flower, the pinecone. There are many species of this tree with different sizes and shapes, but the pinecone is very similar in between species (Figure 60).

As it is visible in the figure 60, pinecones are mostly, as the name says, cone shaped and are composed of "petals" that are wing shaped (see figure 61). The overall shape of the pinecone and the petals were the inspiration to the fashion accessory that is the example design in this dissertation to show one of the many purposes the materials developed can fulfil.

This inspiration came as an "homage" to the waste used and to give emphasis to the fact that the materials developed have in their composition pine wood waste.

5.1.1. Moodboard

As referred before, the object developed is a fashion accessory using the materials designed. It is a small purse that is inspired by pinecones.

To develop the accessory's design, a search was conducted on objects already inspired in pinecones, not only fashion accessories but also other types of objects such as lamps, decoration ornaments, ... to be also used as an inspiration to what the purse could be like.

This objects alongside the pinecones and some other pictures that served as inspiration constitute the Moodboard for this handbag shown in figure 62.



Figure 62: Moodboard for the development of the handbag.

5.1.2. Designing

This previous step led to some ideas and to the development of some sketches. Some initial sketches are in figure 63.

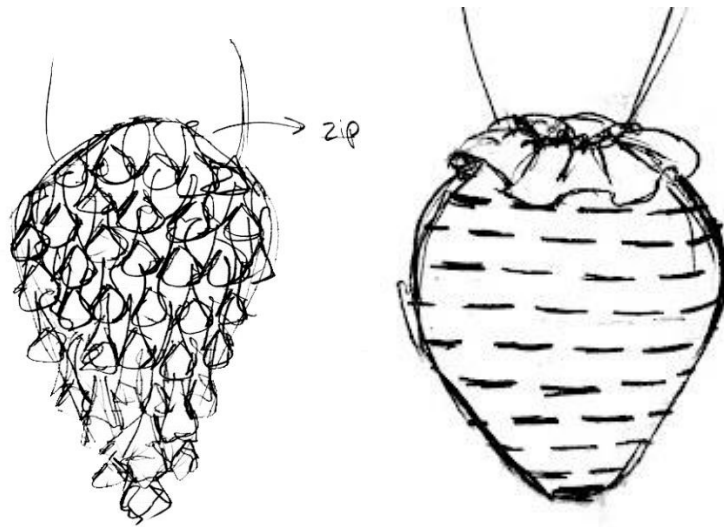


Figure 63: Initial sketches for the project.

Although there were sketches on different ways to produce this purse, from the beginning, the design considered that the “petals” would be hung from the base of the purse, so that it would be similar to the pinecones and so that one of the materials designed would be the base to the petals made from the other designed material.

A design for this purse started to emerge and some experimental models were done to reach a final prototype.

5.2. The petals

To have a successful model the “hung petals” needed to have a “simplified” and “synthesised” shape. A sketch of many possible shapes for the petals was created (figure 64).

There were 3 that were considered the most interesting and were tested as a mockup (number 7, 12 and 16) in a fabric that had similar thickness to the material developed, to understand if they also worked in fabric (figure 65). The finished petals were not possible to reverse to hide the stitching. Glue was also tried, and it didn't look good as well, as it is possible to see in the mockup of petal number 7 presented in figure 65. Another problem with the petals in this size was that they had very little volume and the design had a characteristic volume to enhance the three dimensionality that the real pinecone “petals” have.

Figure 65 also shows another test that was made with the mockups of number 12 and 16 where the stitching was visible but, in this case, the only way to add volume was by stuffing the inside of the petals.

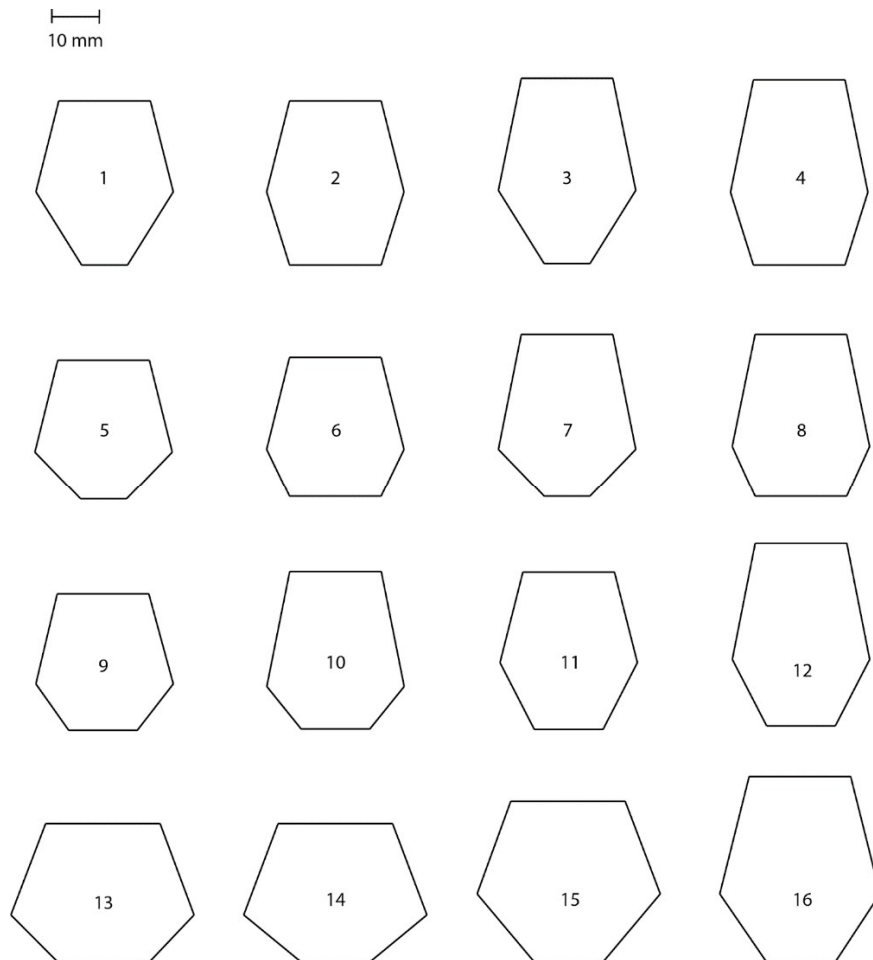


Figure 64: Sketches of the “petals” shapes (not in scale nor real size).

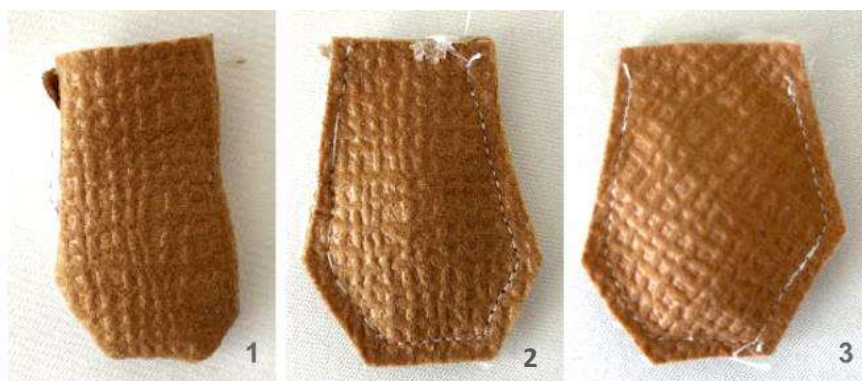


Figure 65: First petals mockups. (1) petal number 7; (2) petal number 12; (3) petal number 16.

These tests were important to understand that the size of the petals was too small, and it would make the stitching very difficult, and the shape wouldn't work. so, the 3 shapes were resized to more 20% and more 50% of the initial size (figure 66). In the sketch phase it was perceived that the option with twice the size would be too big so new mockups were done to test the size with more 20% and check which one of the 3 options of petals worked best and looked as desired (figures 67).

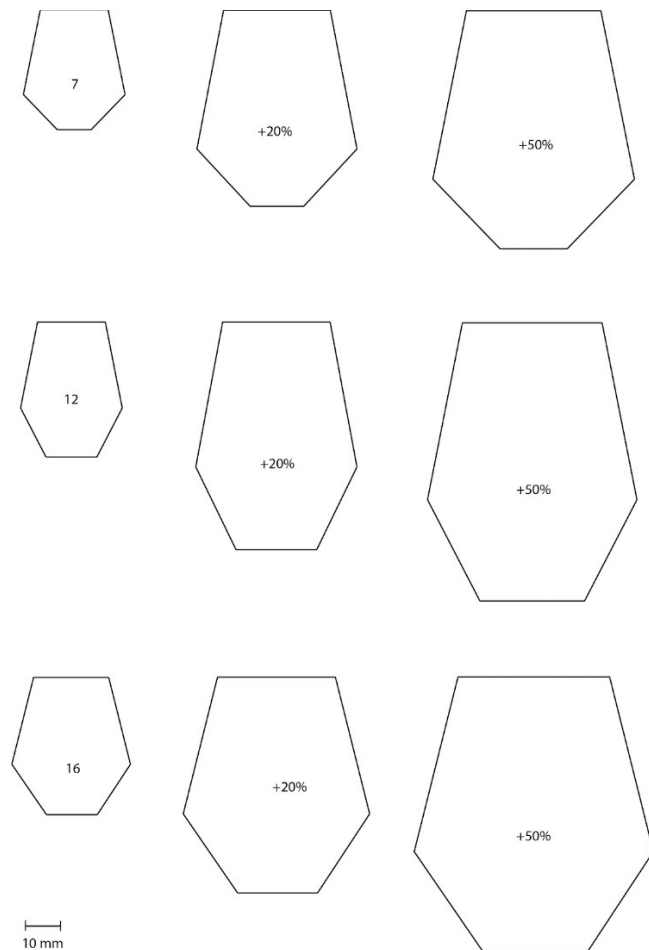


Figure 66: Sketches of the petals number 7, 12 and 16 resized to +20% and + 50% (Not in scale or real size).



Figure 67: Mockups of the petals 7, 12 and 16, respectively, resized to +20%.

The chosen shape was number 7 and this one was tested in samples of the designed material, to make sure that the shape worked in the real material (figure 68).

5.3. The base and shape of the purse.

The “petals” are indeed the main focus of this purse, so the designing started there but, alongside the “petals” testing, it was being tested the overall shape of the purse and how these “petals” would be displayed on it. Sketches were initially made (figure 69) but the shape that turned out to become the base was thought to be the one that resembled the most a pinecone. In this case the petals are not displayed side by side as most of the sketches were designed but they would be interspersed with each other, something similar to the sketch on figure 70.



Figure 68: Mockup of the chosen shape in the designed material.

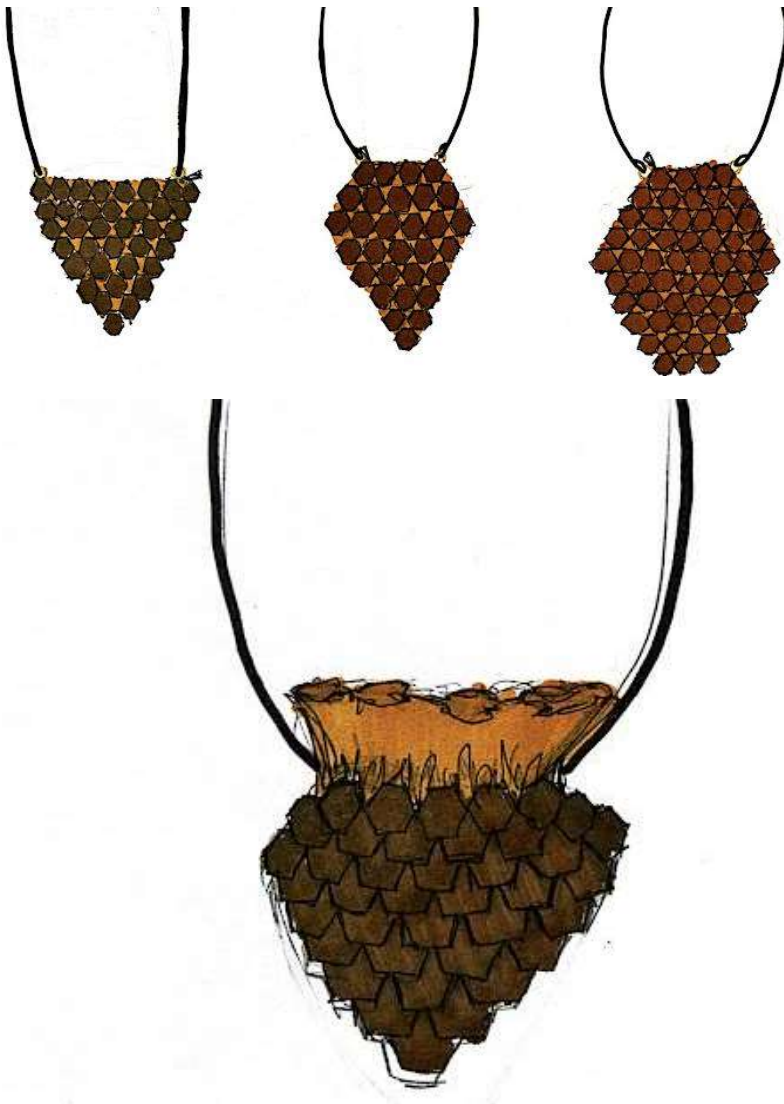


Figure 69: Sketches of the purse formats and the petals arrangement

Figure 70: Final sketch of the purse.

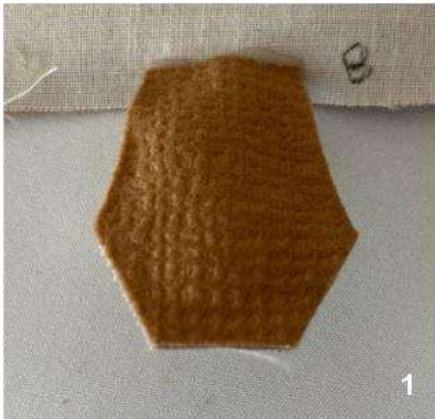


Figure 71: Mockup of the petals if glued to the fabric. (1) front view; (2) side view.



Figure 72: Experimental models of the petals if sewed to the fabric. (1) front view; (2) side view.

As for the way these petals would be attached to the base of the purse, it was experimented in mockups (using the same material used for the mockups of the petals) two different ways to attach them that can be seen in figures 71 and 72. In 71 it is the option where the petals would be glued to the base, using proper fabric glue. It wasn't necessary to even try to glue and display other petals alongside the first just by looking at the results with just one. As it is visible in figure 71, the petals would be too spiky and stiff for the desired result.

The other option, visible in figure 72 is the option where the petals would be sewed to the base and the volume that the petals already have, would make them "spike" enough.

To finish this process, a mockup of the "organisation" of the petals in the base was made. To see if it would work in fabric as desired (figure 73).



Figure 73: Mockup of the organisation of the petals in the base.

5.4. The sewing patterns and final mockup

In order to make sure the drawing of the handbag worked; a final mockup was made. This mockup was made with different materials than the ones that would be used in the final object but using materials with similar thicknesses to make sure that the result in the mockup would transmit a similar behaviour to what would happen in the final prototype.

To begin this process, it was necessary to develop sewing patterns for the components of the purse that can be visible in figures 74, 75 and 76. The final design is composed of repeated pieces of the same 3 patterns: 2 equal pieces for the base of the purse, which correspond to the pattern presented in figure 74; 2 equal pieces that make the upper part of the purse (see pattern in figure 75) and 74 pieces with the drawing of the pattern presented in figure 76 that make the petals.

The final mockup is presented in figure 77. This model was important to test if the handbag worked and if the sewing patterns needed adjustments.

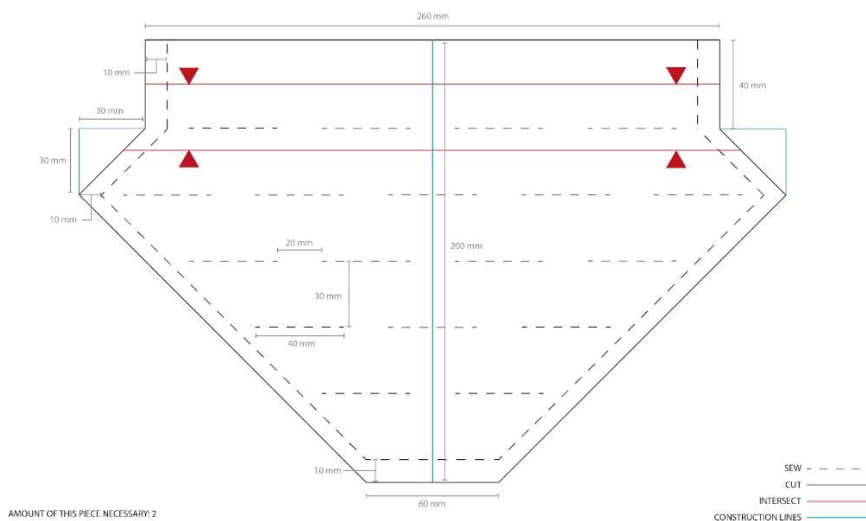


Figure 74: Sewing pattern designed for the pieces that compose the base of the purse (not in scale nor real size).

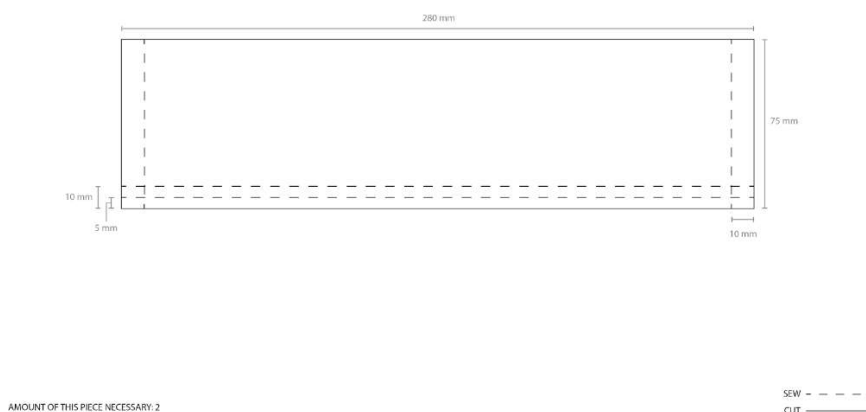


Figure 75: Sewing pattern designed for the pieces that compose the upper part of the purse (not in scale nor real size)

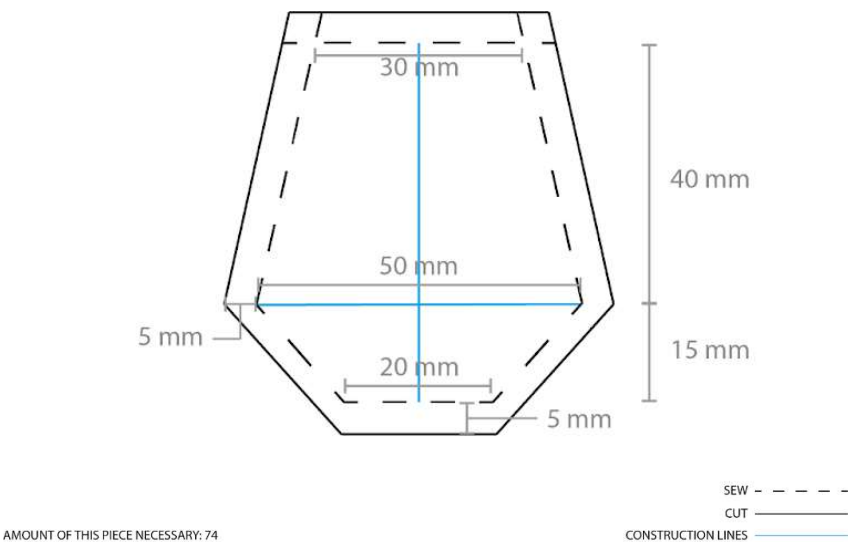


Figure 76: Sewing pattern designed for the pieces that compose the “petals” (not in scale nor real size).



Figure 77: Final mockup of the handbag.

5.5. The prototype

After achieving the final design of the project and confirming it with a mockup, a prototype was then made using the designed coated materials.

As mentioned in the previous subchapter one of the mockup's purposes was to test the sewing patterns and check if they needed adjustments. In fact, the sewing pattern of the base of the handbag underwent some changes that can be seen in figure 78. The other sewing patterns remained as shown above in figures 75 and 76.

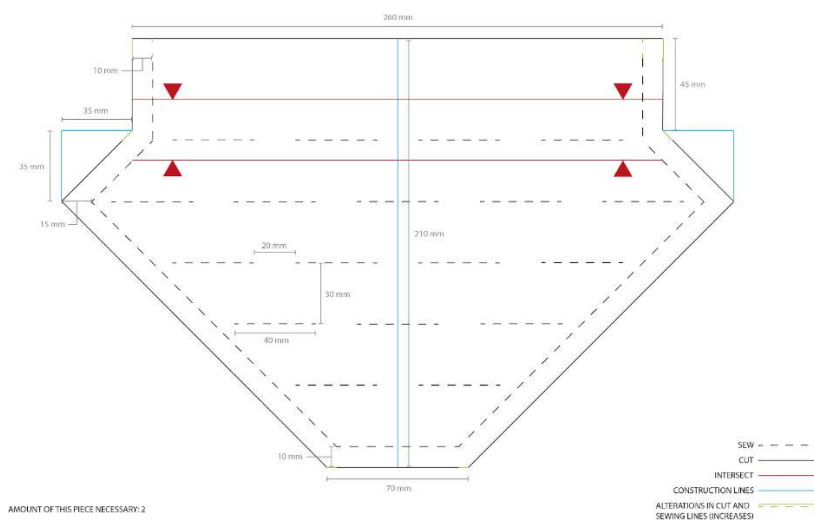


Figure 78: Sewing pattern for the base of the purse updated (not in scale nor real size).

5.5.1. Making of the prototype

To make the prototype of the final design many steps and materials were required. In table 10 all the materials necessary are presented.

Table 10: Materials and tools used to make the prototype.

Designation	Notes & Specifications
Sewing Machine	Pfaff Quilt 1510
Designed coated material	With 7% of pine bark waste
Designed coated material	With 10% of pine sawdust
Cords	-
Sewing threads	-
Sewing pattern for the base	-
Sewing pattern for the upper part	-
Sewing pattern for the “petals”	-
Fabric scissors	-
Stitch remover/ Seam Ripper	-
Graphite pencil	Staedtler B4
Weights	-



Figure 79: Macpi 923.37.1610 laser machine.

Step 1: Laser cutting the pieces for the petals

The material assigned to make the petals was cut by laser in a Macpi 923.37.1610 laser machine like the one shown in figure 79, so that the pieces would all be cut equally and since it required 74 of the same drawing, it would speed up the process instead of manually cutting these (see figure 80).

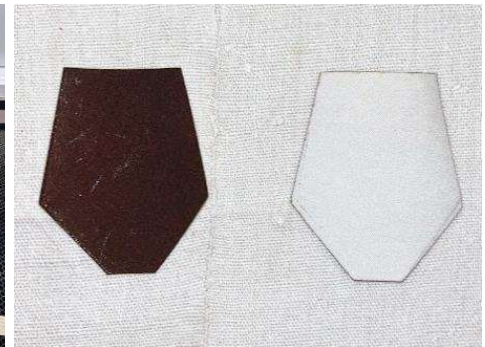
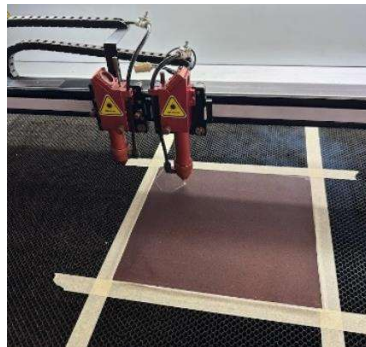


Figure 80: Laser cutting the petals.

Step 2: Stitching the petals

Each petal is composed of two equal pieces using the drawn pattern shown before. In this step, the 74 pieces were paired together to make the 37 petals that compose the purse. The stitching is made on what is the inside of the petals leaving the upper part of the petal unstitched so that it is possible to reverse them to the side of the material that is supposed to be on the outside (see figure 81).

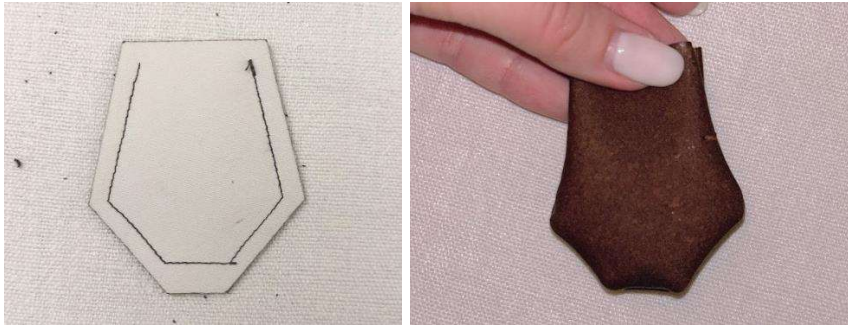


Figure 81: Sewing the petals.

Step 3: Drawing and cutting the base pieces of the purse

In this step, the sewing pattern for the base of the purse was placed on top of the material assigned to this part of the handbag and using the graphite pencil, the shape was drawn in the material. It was also marked where the petals would be stitched in to facilitate the process. The excess material was cut out (see figure 82).

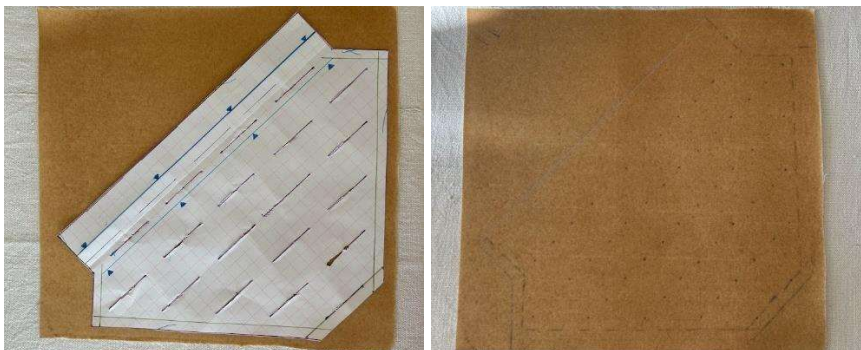


Figure 82: Cutting the base.

Step 4: Sewing the bottom “petal”

The “petal” that makes the bottom of the accessory is placed differently than the rest of the “petals”. It is sewed in between the two pieces that make the base of the bag (figure 83).



Figure 83: Sewing the bottom petal.

Step 5: Sew all the remaining petals

The next step was to sew the remaining “petals” in the places marked in step 3 the way it is shown in picture (1) from figure 84. These are sewn in both of the pieces that make the base of the purse so that the “petals” are in the front and back side of the bag at the end of this step the result is what is shown in picture (2) in figure 84.

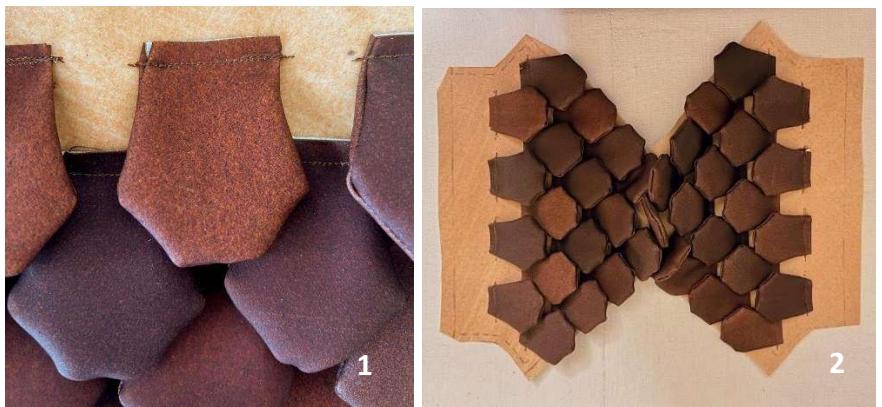


Figure 84: Sewing the remaining petals.

Step 6: Cut and sew the upper part

In this step both pieces that make the upper part of the handbag were cut out (picture (1) in figure 85) from a piece of the material and sewn to the top of the base pieces as shown in picture (2) in figure 85.

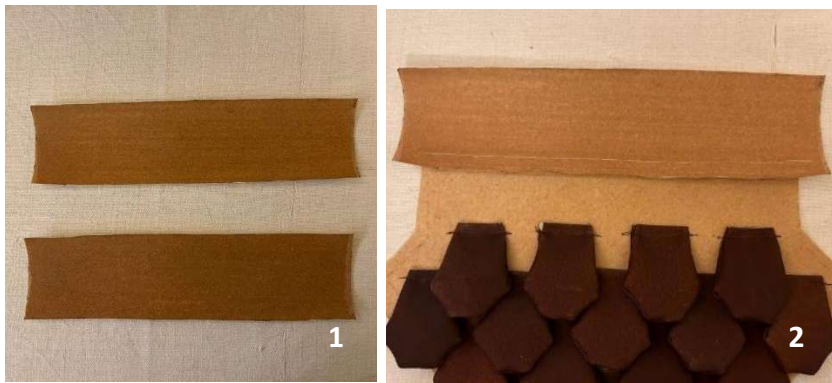


Figure 85: Cut and sewing the upper part.

Step 7: Make the tube where the cord will be inserted

In this step, the remaining visible top part of the base pieces are folded to behind the already sewn upper part until the existing seam that connects these two parts meets the top part of the first row of petals as shown in picture (1) of figure 86. This is sewn in this position about 5 mm below the already existing one that connects the upper parts of the purse to the base parts (see (2) in figure 86). On the inside part it will look like what is presented in picture (3) in figure 86.



Figure 86: Making the tube for the cord.

Step 8: Sew the two bases and upper parts together

For this part, it was necessary to flip what had been done already, in order to have, what is supposed to be the inside of the object, as if it was the outside. This is probably the most difficult step because it is necessary to pin the sides of the base pieces together (like shown in picture (1) of figure 87) but the “petals” that are now temporarily on the inside part, make it difficult because of their volume. The upper parts are also sewn together creating a bag-like shape, leaving the top part open and the result of this step should be something like what is shown in picture (2) of figure 87. At the end of this step, it is necessary to flip the piece so that the “petals” side is again the outside of the handbag and one should have a piece like the one shown in picture (3) of figure 87.

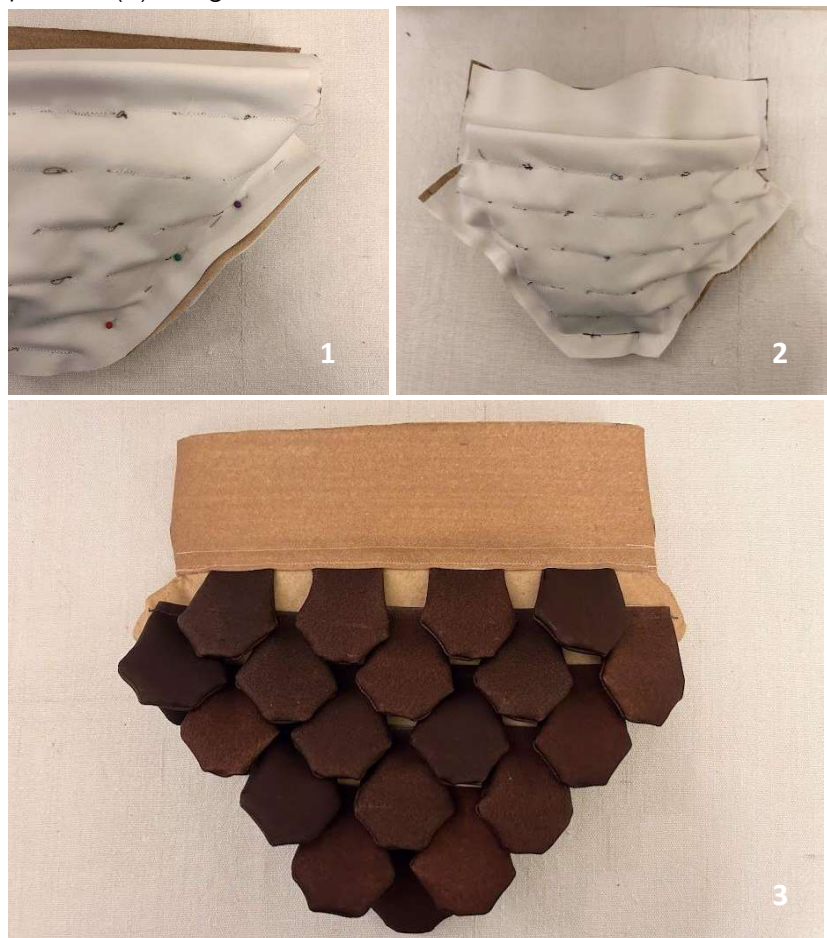


Figure 87: Sew the bases to the upper parts.

Step 9: Making the holes for the cords

In order for the cords that make the “handles” of this bag to go through the tube created in step 7 and still come out of the purse, it is necessary to make two incisions (as shown in figure 88) on each side of the upper part of the accessory.



Figure 88: Making the holes for the cords.

Step 10: Cutting the inside tube

It was necessary to cut triangular shapes on the tube where the cords go through in order for the closing of the bag to be possible. Since the material is thick, when pulling the cords to close the bag it would be very difficult due to the stiffness of the material. These cuts allow for this to be an easier task (figure 89).



Figure 89: Cutting the tube.

Step 11: Insert the cords

For the handles of this design two cords with about 50 cm each are necessary. They are inserted into the tube created in step 7 and the entering point is the holes made in step 9. One cord is supposed to enter one of the holes created in step 9, go through the tube, get out on the other side through the other holes also made in step 9, re-enter the tube but on the other face of the bag and come out through the hole next to the one it initially went through. Basically, make a “U” path on the inside of the tube. The other cord should do the exact same but entering and coming

out on the opposite side to the one that was placed before. The tips of the cords on the same side of the purse are tied together.

This step concludes the making of this handbag and the result is what is shown in picture (1) of figure 90 which shows the purse open, and in picture (2) from figure 90 which shows the bag closed.



Figure 90: Insert the cords; final prototype.

This object, using the two designed coating textiles is an example of one of the many applications that these materials can be used for. It was designed, as said before, as a conceptual piece and, to “honour” the wastes that were incorporated into the coated textiles that make the purse.

These materials could be used for purposes such as upholstery, decorations, lamps, fashion, ...

Due to limitations on sample production, the designed showcasing object couldn't require too much material to make as it would happen in upholstery for example. As so, the choice to make a fashion accessory was mostly based on constraints imposed by the material availability. In the end, these limitations were not considered a problem since the inspiration and “message” of the concept were achieved and considered well represented in the designed handbag.



6.CONCLUSIONS AND FUTURE WORK

6.1. Conclusions

In line with the first objective, this dissertation aimed to create experimental coated materials by utilising pine wood waste obtained from the Chilean wood industry. Applying biomimetic principles, a successful design of a coated material was developed using pine sawdust waste from Chile. The coated material was designed to closely resemble leather in terms of its visual appearance, texture, and specific properties. By employing biomimicry, the research aimed to transform the underutilised pine wood waste into a sustainable alternative to leather, contributing to environmentally conscious material development.

Additionally, the project expanded its scope by incorporating Portuguese pine bark waste, complementing the work conducted in Chile. This inclusion provided a wider perspective on the potential applications of wood waste. The integration of Portuguese pine bark waste enhanced the dissertation's comprehensive understanding of sustainable solutions within the utilisation of wood waste.

To demonstrate the versatility of the designed coated materials, a distinctive handbag, inspired by the form of pinecones, was developed. This concept not only acknowledges the incorporation of waste materials into the coated materials but also serves as a tangible illustration of their diverse applications. By showcasing this unique object, the dissertation highlights the potential benefits and contributions of these coated materials in various domains, emphasising their value in sustainable design and the effective utilisation of resources.

6.2. Final considerations, limitations and future work

As previously mentioned, it is important to note that the designed coated materials are experimental in nature, and only preliminary tests have been conducted. Consequently, further investigation and improvements are necessary to enhance their performance. Several flaws have been identified that can be addressed to enhance the material, as evidenced in the testing chapter. For instance, it has been observed that the material's resistance to both natural and artificial light could be improved.

One of the proposed tests in this dissertation was the surface touch Kawabata testing. Unfortunately, the required equipment for conducting these tests was not available at CITEVE, except for one component of the Kawabata set of tests, known as KES-FB4. Although this equipment was operational and capable of conducting the test, it did not generate a comprehensive report. Instead, it only provided a graphical representation of the test on the screen, without the ability to export the necessary information. Consequently, the data required to draw conclusive results from this particular test could not be obtained.

As part of future work, it would be beneficial to conduct thorough testing on the coated materials, including the Kawabata surface touch set of tests. This would provide a better understanding of their properties and identify any additional improvements necessary to ensure the material's viability for industrial application.

It is important to acknowledge that this dissertation required knowledge in multiple areas, including chemistry, in which the author had limited proficiency. This limitation was identified as a challenge in developing a practical solution within the context of this dissertation.

In spite of these limitations the work developed is going to be included as a chapter in the book “Biomimetics, Biodesign and Bionics: Technological Advances Towards a Sustainable Development” which is tabled to be published by November 2023. CITEVE has also showed interest in submitting the final object to the iTechStyle Green Circle – Sustainability Showcase, an initiative that promotes in international markets Portuguese textile products that are related to sustainability and circular design. This work will also be submitted to the awards of ‘Dia Mundial dos Materiais’ which will take place in November 2023.

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8. APPENDIX

EXPERIMENT #PT01

OBJECTIVE: Test different solutions to see which one seemed more interesting to begin with.

DATE: 22/02/2023

TEXTILE:

TEXTILE SUBSTRATE	Woven Fabric Cotton + Polyester
	Paper

TESTS:

DESCRIPTION	AMOUNT %	TESTED IN PAPER	TESTED IN FABRIC
POLYMER 5 + Pine bark not dried	8g + 11	Yes	Yes
POLYMER 2 + Pine bark not dried	8g + 11	Yes	Yes
POLYMER 4 + POLYMER 3 + Pine bark not dried	8g + 11	Yes	Yes
POLYMER 5 + Dried pine bark	8g + 11	Yes	No
POLYMER 2 + Dried pine bark	8g + 11	Yes	No
POLYMER 4 + POLYMER 3 + Dried pine bark	8g + 11	Yes	Yes

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
Application Machine	W. Mathis AG
Brush	-
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES
DRYING	100 °C/ 5 min.
CURING	150 °C/ 5 min.

NOTES:

- This was the first test to be done with the pine bark waste.
- Both the dried pine bark and the not dried pine bark waste were tested.
- Very small amounts of pastes were made to test different polymers with the waste (table "Tests" lists the variations that were carried out).
- The proportion of wood residue in these pastes is 11%.
- These pastes are only composed of polymer and waste. No other component was added.
- These small amounts of pastes were applied with a brush in plasticised paper and some, also in a woven fabric.
- They were dried for 5 minutes at 100°C.
- They were also calendared for 5 minutes for 150 °C.

CONCLUSIONS/OBSERVATIONS/RESULTS:

- POLYMER 5 + Pine bark not dried: this polymer is not bio based which makes it a less desired material to choose. Also, the results in either paper and fabric feel very stiff and rough, but it doesn't crack.
- POLYMER 2 + Pine bark not dried: looked promising, a little rough but, less than with POLYMER 5. It can be the % of waste added that is causing it to have a rough feel.
- POLYMER 4 + POLYMER 3 + Pine bark not dried: it is the paste that created more lumps. It results in a very stiff and harsh surface.
- POLYMER 5 + Dried pine bark: to the touch it feels smoother however, the surface cracked.
- POLYMER 2 + Dried pine bark: Result very similar to the result with the not dried pine bark.
- POLYMER 4 + POLYMER 3 + Dried pine bark: Result very similar to the result with the not dried pine bark.

EXPERIMENT #PT02

OBJECTIVE: Tests with the polymer POLYMER 2

DATE: 01/03/2023

TEXTILE:

TEXTILE SUBSTRATE	Woven Fabric polyester + cotton (thick)
	Knitted Fabric 100% cotton

COATING FORMULATION: 100g

	DESIGNATION	AMOUNT (g)	%
POLYMER	POLYMER 2	94.3	94.3
WASTE	Pine Bark	5	5
ADDITIVES: Crosslinker	CROSSLINKER 1	0.7	0.7
Thickener (diluted as instructed)	THICKENER 4 + water (1/1)	q.b.	-

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
Scale	PG 5002 Deltarange
Mixer	Techmatic S2
pH measurer	HANNA
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Calender	Macpi 553.37-9124 FLAT BONDING PRESS
Calender paper	Sappi – v/c universal carrara (971)
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	
DRYING	100°C/ 5 min./ 1700 rpm*	Each layer dried separately
CURING	150°C/ 5 min./ 1700 rpm*	After all layers applied
LAYERS	3x 0.0mm	-
CALENDERING	140°C/ 30 sec./ 3 bars	-

*Fan Speed

OTHER INFO:

	VALUES	NOTES
VISCOSITY	6.80 Pa.s (6800 cps)	100 rpm Torque: 68% s 06*
pH	6.46	-

* S means Spindle and the number is the identifier of the spindle.

NOTES:

- The polymer was mixed with waste then crosslinker was added then thickener was added (drop by drop) until visually it was less liquid, sticking to the spoon. The procedure was incorrect: the crosslinker should have been added first.
- The thickener has to be diluted in water: equal parts of water and thickener were mixed in a separate cup.
- The measurement of pH and viscosity was done as soon as the paste was ready to be applied.
- This paste was applied in 2 different samples of textile: a Woven Fabric (1) and a Knitted Fabric. (2)
- The 3rd layer of paste in (1) was applied only in half of the textile sample.
- The calendaring was done in two different steps of the process for the different samples. In (1) it was done after curing and in (2) it was done before curing.

CONCLUSIONS/OBSERVATIONS/RESULTS:

(1) Woven Fabric

- This paste, after 3 layers of application, curing, and before the embossing/calendering has a rough texture.
- With the calendaring it acquires a different texture, softer and smooth.
- It acquires a very hard and stiff surface, but it doesn't crack.
- The embossing done in the calendaring with a textured paper is hardly perceivable since it has been done after curing.

(2) Knitted Fabric

- In this sample the embossing textured paper and calendaring were done before curing so the texture of the embossing is more perceptive.
- It is more malleable.
- It didn't transfer (as it was expected to).
- It didn't crack.

EXPERIMENT: #PT03

OBJECTIVE: Tests using the polymer POLYMER 2 without crosslinker + control paste to compare texture.

DATE: 06/03/2023

TEXTILE:

TEXTILE SUBSTRATE	Woven Fabric polyester + cotton (thin)
	Knitted Fabric 100% cotton

COATING FORMULATION: 300g

	DESIGNATION	AMOUNT (g)	%
POLYMER	POLYMER 2	285	95
WASTE	Pine Bark	15	5
ADDITIVES: Thickener	THICKENER 3	q.b	-

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
Scale	PG 5002 Deltarange
Mixer	Techmatic S2
pH measurer	ImoLab pH7150
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	
DRYING	100 °C/ 5 min./ 1300 rpm*	Each layer dried separately
CURING	150 °C/ 5 min./ 1300 rpm*	After all layers applied
LAYERS	3x 0.0mm	-

*Fan Speed

OTHER INFO:

	VALUES	NOTES
VISCOSITY	6.34 Pa.s (6340 cps)	50 rpm Torque: 79.3% s 05*
pH	6.59	-
Control Paste: viscosity/ pH	5.40Pa.s /8.067	50 rpm Torque: 67.5 s05*

* S means Spindle and the number is the identifier of the spindle.

NOTES:

- The polymer was mixed with waste then thickener was added (drop by drop) until visually it was less liquid, sticking to the spoon.
- The measurement of pH and viscosity was made as soon as the paste was ready to be applied.
- A Control Paste was made, with the same ingredients excluding the residue (100% polymer + q.b of thickener. Parameters of application were the same.) alongside the paste using residue for comparison purpose.
- These pastes were applied in 2 different samples of textile: a Woven Fabric and a Knitted Fabric.

CONCLUSIONS/OBSERVATIONS/RESULTS:(1) Woven Fabric

- The paste using waste and this polymer continues to result in a very hard and stiff surface.
- In this fabric, the control paste transferred but the one with waste didn't.

(2) Knitted Fabric

- The paste using waste and this polymer continues to result in a very hard and stiff surface, although, in this fabric sample, it is a little bit less hard, since it is a softer substrate.
- None of the pastes transferred but the control paste acquired bubbles in the surface.

EXPERIMENT #PT04

OBJECTIVE: Tests with polymer POLYMER 4

DATE: 06/03/2023

TEXTILE:

TEXTILE SUBSTRATE	Woven fabric polyester + cotton (thin)
	Knitted fabric 100%cotton
	Elastic knitted fabric

COATING FORMULATION: 300g

	DESIGNATION	AMOUNT (g)	%
POLYMER	POLYMER 4	285	95
WASTE	Pine Bark	15	5
ADDITIVES: Thickener	THICKENER 1	q.b	-

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
scale	PG 5002 Deltarange
Mixer	Techmatic S2
pH measurer	InoLab pH7110
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	
DRYING	100 °C/ 5 min./ 1300 rpm*	Each layer dried separately
CURING	150 °C/ 5 min./ 1300 rpm*	After all layers applied
LAYERS	3x 0.0 mm	-

*Fan Speed

OTHER INFO:

	VALUES	NOTES
VISCOSITY	8.85 Pa.s (8850 cps)	60 rpm Torque: 53.1% s06*
pH	7.798	-
Control Paste: viscosity/ pH	7.55 Pa.s / not measured	100rpm Torque: 75.5 s06*

* S means Spindle and the number is the identifier of the spindle.

NOTES:

- The polymer was mixed with waste then thickener was added (drop by drop) until visually it was less liquid, sticking to the spoon.
- The measurement of pH and viscosity was as soon as the paste was ready to be applied.
- A Control Paste was made, with the same ingredients excluding the residue (100% polymer + q.b of thickener. Parameters of application were the same.) alongside the paste using residue for comparing purposes.
- These pastes were applied in 3 different samples of textile, one woven fabric (1) and two different types of knitted fabric, one without elastic (2) and one with elastic in its composition (3).

CONCLUSIONS/OBSERVATIONS/RESULTS:

- The textures of these samples was better than the ones in the test #PT03 for all the different types of fabric in which the pastes were applied.
- They all were more promising than the ones in #PT03: less stiff surfaces, especially in the knitted fabrics samples.

EXPERIMENT #PT04.1

OBJECTIVE: Tests with polymer POLYMER 4 with incorporation of crosslinker in the paste to compare with the results with the same paste formulation without crosslinker (#PT04).

DATE: 06/03/2023

TEXTILE:

TEXTILE SUBSTRATE	Elastic Knitted Fabric
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COATING FORMULATION: 150g

	DESIGNATION	AMOUNT (g)	%
POLYMER	POLYMER 4	141.45	94.3
WASTE	Pine Bark	7.5	5
ADDITIVES: Thickener	THICKENER 1	q.b	-
Crosslinker	CROSSLINKER 2	1.5	0.7

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
scale	PG 5002 Deltarange
Mixer	Techmatic S2
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	
DRYING	100 °C/ 5 min./ 1300 rpm*	Each layer dried separately
CURING	150 °C/ 5 min./ 1300 rpm*	After all layers applied
LAYERS	3 layers all in 0.0mm	-

*Fan Speed

OTHER INFO:

	VALUES	NOTES
VISCOSITY	5.96 Pa.s (5960 cps)	100 rpm Torque: 59.6% s06*

* S means Spindle and the number is the identifier of the spindle.

NOTES:

- Mixed polymer with crosslinker then waste and finally, thickener (drop by drop) until visually it was less liquid, sticking to the spoon.
- The measurement of viscosity was as soon as the paste was ready to be applied.
- This paste was applied in an elastic knitted fabric.

CONCLUSIONS/OBSERVATIONS/RESULTS:

- Not much different texture and surface than the paste in #PT04 applied in the same fabric.

EXPERIMENT #PT05

OBJECTIVE: Tests with polymer POLYMER 4 with incorporation of emulsifier applied in 2 different fabrics.

DATE: 14/03/2023

TEXTILE:

TEXTILE SUBSTRATE	Elastic Knitted Fabric
	Woven fabric polyester+cotton (thick)

COATING PASTE FORMULATION: 150g

	DESIGNATION	AMOUNT (g)	%
POLYMER	POLYMER 4	142.45	95
WASTE	Pine Bark	7.5	5
ADDITIVES: Thickener	THICKENER 1	q.b	-
Emulsifier	EMULSIFIER 1	0.3*	-

*Emulsifier is added to the paste, but it is not accounted for in the percentages. Its relation is 2g per 1kg of paste.

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
scale	PG 5002 Deltarange
Mixer	Techmatic S2
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	
DRYING	100 °C/ 5 min./ 2300 + 1300 rpm*	
CURING	150 °C/ 5 min./ 2300 + 1300 rpm*	After all layers applied
LAYERS	3 layers: 2x 0.0mm + 1x 0.1mm	-

*Fan Speed

OTHER INFO:

	VALUES	NOTES
VISCOSITY	6.01 Pa.s (6010 cps)	60 rpm Torque: 90.1% s05*

* S means Spindle and the number is the identifier of the spindle.

NOTES:

- Polymer was mixed with emulsifier, then waste and finally, thickener was added (drop by drop) until visually it was less liquid, sticking to the spoon.
- The measurement of viscosity was made as soon as the paste was ready to be applied.
- This paste was applied in an elastic knitted fabric and a woven fabric.
- It was necessary to add distilled water to this paste to adjust the thickness, since too much thickener was added .
- These samples have two layers with 0.0mm and one in 0.1mm.

CONCLUSIONS/OBSERVATIONS/RESULTS:

- The results in these samples were unsatisfactory, although the reason wasn't clear.
- This paste showed lumps of waste. The cause wasn't determined.
- During the process of drying, the colour was much darker than the other samples from previous tests made with the same polymer and the same percentage of waste. The cause wasn't determined.
- Were the measurements done wrongly by mistake and that is why it acquired such darker colour?
- Was the fact that the last layer was a bit higher the reason the sample was darker?
- Did the residue gain humidity and that is why it lumped in the paste?
- Was there a problem with viscosity? Was it too high?

(1) Elastic Knitted Fabric

- This sample shows better results than the woven fabric one. Still, it shows lumps in the surface. The emulsifier should have helped in this matter, but it didn't.

(2) Woven Fabric

- There are visible lumps on the surface.
- The woven fabric was the first in which the paste was applied, there was an error and the fan speed in drying and curing was 2300 rpm. This was determined to NOT be the cause of such bad results since a new sample was made in the same fabric using a fan speed of 1300rpm and the results were as bad if not even worse.
- These samples showed bubbles of air right after the first layer drying. In the sample using 1300rpm in the fan speed, it even peeled. The cause wasn't determined.

EXPERIMENT #PT05.1

OBJECTIVE: Tests with polymer POLYMER 4 with incorporation of emulsifier and crosslinker applied in 2 different fabrics to compare with the results in #PT05.

DATE: 14/03/2023

TEXTILE:

TEXTILE SUBSTRATE	Woven fabric polyester+cotton (thick)
	Elastic Knitted Fabric

COATING FORMULATION: 150g

	DESIGNATION	AMOUNT (g)	%
POLYMER	POLYMER 4	141.5	94.3
WASTE	Pine Bark	7.5	5
ADDITIVES: Thickener	THICKENER 1	q.b	-
Emulsifier	EMULSIFIER 1	0.3*	-
Crosslinker	CROSSLINKER 2	1.5	0.7
Topcoat	TOPCOAT 1 + POLYMER 1 **	5	-

*Emulsifier is added to the mixture according to its quantity. The relation is 2g of emulsifier for each 1 kg of paste.

**equal parts of each solution. Topcoat is a layer added after on its own. Not mixed in the paste.

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
scale	PG 5002 Deltarange
Mixer	Techmatic S2
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	
DRYING	100 °C/ 5 min./ 2300 + 1300 rpm*	Each layer dried separately
CURING	150 °C/ 5 min./ 2300 + 1300 rpm*	After all layers applied
LAYERS	3 layers: 2x 0.0mm + 1x 0.1mm	-

*Fan Speed

OTHER INFO:

	VALUES	NOTES
VISCOSITY	6.29 Pa.s (6290 cps)	60 rpm Torque: 94.4 s05*

* S means Spindle and the number is the identifier of the spindle.

NOTES:

- Order of mixing was polymer, then emulsifier and crosslinker mixed in at the same time, then waste was added and mixed, then thickener drop by drop until visually it was less liquid, sticking to the spoon.
- The measurement of viscosity was as soon as the paste was ready to be applied.
- This paste was applied in 2 different types of fabric: a woven fabric (1) and an elastic knitted fabric (2).
- It was necessary to add distilled water to this paste to control the thickness since it was added to much thickener.
- In this samples it was applied two layers with 0.0mm and one with 0.1mm.

CONCLUSIONS/OBSERVATIONS/RESULTS:

- The results in this samples were very unsatisfactory, although the reason wasn't clear.
- This paste showed lumps of waste.
- During the process of drying the colour was very much darker than the other samples from previous tests made with the same polymer and the same percentage of residue. The cause wasn't determined.
- Were the measurements done wrongly by mistake and that is why it acquired such darker colour?
- Did the fact that the last layer is a bit higher had a role in the sample being darker?
- Did the residue gain humidity and that is why it lumped in the paste?
- Was there a problem with viscosity? Was it too high?

(1) Woven Fabric

- Lumps in the surface are visible.
- In the woven fabric sample, as it was the first in which we applied the paste, there was an error and the fan speed in drying and curing was in 2300 rpm. This was determined to NOT be the cause of such bad results as it was made a new sample in the same fabric using a fan speed of 1300rpm and the results were as bad if not even worse.
- This samples showed bubbles of air right after the first layer drying. In both samples, the one in which 2300 rpm were used and the one where it was used 1300rpm in the fan speed, the coating peeled. The cause wasn't determined.

(2) Elastic Knitted Fabric

- This sample shows better results than the woven fabric one. Still, it shows lumps in the surface. The emulsifier should have helped in this matter, but it didn't.
- In part of this sample, it was applied a top coat. It acquired a smother and mate surface.

EXPERIMENT #PT05.2

OBJECTIVE: Tests with polymer POLYMER 4 with incorporation of crosslinker, not using emulsifier and using pine bark waste previously dried. Applied in 2 different fabrics to compare with the results in #PT05 and #PT05.1.

DATE: 14/03/2023

TEXTILE:

TEXTILE SUBSTRATE	Woven fabric polyester + cotton (thick)
	Elastic Knitted Fabric

COATING FORMULATION: 150g

	DESIGNATION	AMOUNT (g)	%
POLYMER	POLYMER 4	141.5	94.3
WASTE	Pine Bark (previously dried)	7.5	5
ADDITIVES: Thickener	THICKENER 1	q.b	-
Crosslinker	CROSSLINKER 2	1.5	0.7
Topcoat	TOPCOAT 1 + POLYMER 1 XTP*	5	-

*Equal parts of each solution. Topcoat is a layer added after, on its own. Not mixed in the paste.

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
scale	PG 5002 Deltarange
Mixer	Techmatic S2
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	
DRYING	100 °C/ 5 min./ 2300 + 1300 rpm*	Each layer dried separately
CURING	150 °C/ 5 min./ 2300 + 1300 rpm*	After all layers applied
LAYERS	3 layers: 2x 0.0mm + 1x 0.1mm	-

*Fan Speed

OTHER INFO:

	VALUES	NOTES
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VISCOSITY	6.21 Pa.s (6210 cps)	100 rpm Torque: 62.1 s06*
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* S means Spindle and the number is the identifier of the spindle.

NOTES:

- Order of mixing was polymer, then crosslinker mixed, then waste was added and mixed, then thickener drop by drop until visually it was less liquid, sticking to the spoon.
- The measurement of viscosity was made as soon as the paste was ready to be applied.
- This paste was applied in 2 different types of fabric: a woven fabric (1) and an elastic knitted fabric (2).
- It was necessary to add distilled water to this paste to adjust the thickness since too much thickener was added.
- In this samples two layers were applied, one with 0.0mm and one with 0.1mm.
- In this test the waste used was previously dried to check the difference between the results with dried waste and not dried waste.

CONCLUSIONS/OBSERVATIONS/RESULTS:

- The results in these samples were unsatisfactory, although the reason wasn't clear.
- This paste showed lumps of waste.
- During the process of drying the samples the colour was much darker than the other samples from previous tests made with the same polymer and the same percentage of waste. The cause wasn't determined.
- Were the measurements done wrongly by mistake and that is why it acquired such darker colour?
- Was the fact that the last layer was a bit higher the reason for the darker colour of the sample ?
- Did the waste gain humidity and that is why it lumped in the paste?
- Was there a problem in viscosity? Was it too high?
- The colour was found to be slightly darker than the samples using undried waste.

(1) Woven Fabric

- Lumps in the surface are visible.
- In the woven fabric sample, as it was the first in which we applied the paste, there was an error and the fan speed in drying and curing was in 2300 rpm. This was determined to NOT be the cause of such bad results as a new sample was made in the same fabric using a fan speed of 1300rpm and the results were as bad if not even worse.
- These samples showed bubbles of air right after the first layer drying. In both samples, the one in which 2300 rpm was used and the one where 1300rpm was used in the fan speed, the coating peeled. The cause wasn't determined.

(2) Elastic Knitted Fabric

- This sample shows better results than the woven fabric one. Still, it shows lumps in the surface.
- In part of this sample, a topcoat was applied. It acquired a smother and mate surface.

EXPERIMENT #PT06

OBJECTIVE: The objective with this test, alongside test #PT06.1, was to try similar pastes to the ones used in previous tests, that went wrong (#PT05; #PT05.1; #PT05.2) to try to understand what went wrong. In this test the same recipe as in #PT05.2 is used. At the same time this test serves to experiment with different amounts and thicknesses of layers.

DATE: 20&21/03/2023

TEXTILE:

TEXTILE SUBSTRATE	Woven fabric polyester + cotton (thick)
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COATING FORMULATION: 150g

	DESIGNATION	AMOUNT (g)	%
POLYMER	POLYMER 4	141.5	94.3
WASTE	Pine Bark (previously dried)	7.5	5
ADDITIVES: Thickener	THICKENER 1	q.b	-
Crosslinker	CROSSLINKER 2	1.5	0.7

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
scale	PG 5002 Deltarange
Mixer	Techmatic S2
Application Machine	Mathis AG LABCOATER type «LTE-S».
Viscometer	Brookfield DVE Viscometer
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	-
DRYING	100 °C/ 5 min./ 1300 rpm*	Each layer dried separately
CURING	150 °C/ 5 min./ 1300 rpm*	After all layers applied
LAYERS	3 layers: 2x 0.0mm + 1x 0.1mm 4 layers: 2x 0.0mm + 2x 0.1mm	Sample 1 Sample 1.1

*Fan Speed

OTHER INFO:

	VALUES	NOTES
VISCOSITY	4.64 Pa.s (4640 cps)	100 rpm Torque: 46.4 s06*

* S means Spindle and the number is the identifier of the spindle.

NOTES:

- The order of mixing was polymer, then crosslinker mixed, then residue was added slowly, not all at the same time, as usually done, while the paste was mixing continuously, then thickener was added drop by drop until visually it was less liquid, sticking to the spoon.
- The measurement of viscosity was made as soon as the paste was ready to be applied.
- This paste was applied in 2 pieces of the same thick woven fabric, this samples are called PT06(1) and PT06(1.1). Sample PT06 (1) was done in 20/03/2023 and (1.1) in 21/03/2023.
- It was necessary to add distilled water to this paste to adjust the thickness since too much thickener was added.
- In the sample PT06(1) two layers were applied, one with 0.0mm and one with 0.1mm (20/03/2023) and in the sample PT06(1.1) four layers were applied, two with 0.0mm and two with 0.1mm (21/03/2023).
- In this test, previously dried residue was used.

CONCLUSIONS/OBSERVATIONS/RESULTS:

- Two samples were made with the same paste on different days to test if the problem was in the paste drying or oxidation or going through any chemical process overnight. There wasn't any evidence of this being a problem. The paste behaved equally in both days/applications.
- This test was done at the same time as test #PT06.1, with the only difference that the two pastes #PT06.1 had an emulsifier and this one did not.
- Both samples PT06(1) and PT06(1.1) show a glossier surface when compared to the results in #PT06.1.
- Since the paste was behaving equally, in the second application, #PT06(1.1), a fourth layer was applied to test its response, surface, look,... It didn't crack and it normally is a stiffer and darker surface, although this change of colour isn't as noticeable as it happens in the test #PT06.1 but the colour in this test is initially already darker than in #PT06.1.
- In this paste the viscosity was decreased to make sure it wasn't a problem.

EXPERIMENT #PT06.1

OBJECTIVE: The objective with this test, alongside test #PT06, was to try similar pastes to the ones used in previous tests, that went wrong (#PT05; #PT05.1; #PT05.2) to try to understand what went wrong. In this test it is used the same recipe as in #PT05.1 except for the waste that is dried while in #PT05.1 it was not. At the same time this test serves to experiment with different amounts and thicknesses of layers.

DATE: 20 e 21/03/2023

TEXTILE:

TEXTILE SUBSTRATE	Woven fabric polyester+cotton (thick)
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COATING FORMULATION: 150g

	DESIGNATION	AMOUNT (g)	%
POLYMER	POLYMER 4	141.5	94.3
WASTE	Pine Bark (previously dried)	7.5	5
ADDITIVES: Thickener	THICKENER 1	q.b	-
Crosslinker	CROSSLINKER 2	1.5	0.7
Emulsifier	EMULSIFIER 1	0.3*	-

*Emulsifier is not accounted for in the percentages of the paste. Its relation is 2g of emulsifier for each 1 kg of paste.

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
scale	PG 5002 Deltarange
Mixer	Techmatic S2
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	
DRYING	100 °C/ 5 min./ 1300 rpm*	Each layer dried separately
CURING	150 °C/ 5 min./ 1300 rpm*	After all layers applied
LAYERS	3 layers: 2x 0.0mm + 1x 0.1mm 4 layers: 2x 0.0mm + 2x 0.1mm	Sample 2 Sample 2.1

*Fan Speed

OTHER INFO:

	VALUES	NOTES
VISCOSITY	5.91 Pa.s (5910 cps)	100 rpm Torque: 59.1 s06*

* S means Spindle and the number is the identifier of the spindle.

NOTES:

- Order of mixing was polymer, then crosslinker and emulsifier mixed at the same time, then residue was added slowly, not all at the same time, as usually done, while the paste was mixing continuously, then thickener was added drop by drop until visually it was less liquid, sticking to the spoon.
- The measurement of viscosity was made as soon as the paste was ready to be applied.
- This paste was applied in 2 pieces of the same thick woven fabric, this samples are called #PT06.1(2) and #PT06.1(2.1). Sample #PT06.1(2) was done in 20/03/2023 and #PT06.1(2.1) in 21/03/2023.
- In the sample #PT06.1(2) two layers with 0.0mm and one with 0.1mm were applied (20/03/2023) and in the sample #PT06.1(2.1) two layers with 0.0mm and two with 0.1mm were applied (21/03/2023).
- In this test, previously dried residue was used.

CONCLUSIONS/OBSERVATIONS/RESULTS:

- Two samples were made with the same paste on different days to test if the problem was in the paste drying or oxidation or going through any chemical process overnight. There wasn't any evidence of this being a problem. The paste behaved equally in both days/applications.
- This test was done at the same time as test #PT06, with the only difference that the two pastes #PT06 didn't have an emulsifier and this one had.
- Both samples #PT06.1(2) and #PT06.1(2.1) show a less glossy surface when compared to the results in #PT06.
- Since the paste was behaving equally, in the second application, #PT06.1(2.1), another layer was applied to test its response, surface, look, ... It didn't crack and it normally is a stiffer and darker surface. The difference between the colour is more noticeable when compared to the results in #PT06, although, the colour in #PT06 is darker.
- In this paste the viscosity was decreased to make sure it wasn't a problem.

EXPERIMENT #PT07

OBJECTIVE: With this test the objective was to try an increase of the residue percentage using the same products in the paste as used in #PT06 as well as compare results with the tests #PT08.1 and #PT08.2 made alongside.

DATE: 04/04/2023

TEXTILE:

TEXTILE SUBSTRATE	Polyester woven fabric
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COATING FORMULATION: 100g

	DESIGNATION	AMOUNT (g)	%
POLYMER	POLYMER 4	92.3	92.3
WASTE	Pine Bark (previously dried)	7	7
ADDITIVES: Thickener	THICKENER 1	q.b	-
Crosslinker	CROSSLINKER 2	0.7	0.7

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
scale	PG 5002 Deltarange
Mixer	Techmatic S2
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	-
DRYING	100 °C/ 5 min./ 1300 rpm*	Each layer dried separately
CURING	150 °C/ 5 min./ 1300 rpm*	After all layers applied
LAYERS	2x 0.0mm	-

*Fan Speed

OTHER INFO:

	VALUES	NOTES
VISCOSITY	5.60 Pa.s (5600 cps)	100 rpm Torque: 56.0 s06*

* S means Spindle and the number is the identifier of the spindle.

NOTES:

- Order of mixing was polymer, then crosslinker mixed, then waste was added slowly, not all at the same time, as previously done, while the paste was mixing continuously,

then thickener was added drop by drop until visually it was less liquid, sticking to the spoon.

- The measurement of viscosity was made as soon as the paste was ready to be applied.
- In the sample two layers with 0.0mm were applied, as a new amount of waste was being tested.
- In this test, previously dried residue was used.
- The fabric used in this test is a different type of polyester and cotton woven fabric. It is less thick than the one that was being used until this test.

CONCLUSIONS/OBSERVATIONS/RESULTS:

- The results in this test were interesting: it didn't crack and it isn't a very stiff surface, although it presents some lumps on the surface that resulted in some "strings" being visible.
- The surface is somewhat rough, but it seems a more even surface when compared with the results in #PT06 (excluding the places where there are "strings").
- It was considered an interesting path to pursue so from this point on, the tested pastes using pine bark started to have 7% of waste instead of 5%.
- It is believed that the change of the substrate (fabric) also led to a better result, at least in terms of reducing the stiffness of the surface since it is a less thick fabric in itself.

EXPERIMENT #PT08.1 and #PT08.2

OBJECTIVE: The objective of this test was to use ball milled waste instead of what was used until here that was shredded in a miller. The behaviour of the waste when ball milled was assessed since it produced smaller particle size which could make the coating more even and smoother. In these tests the goal was also to try the 5% and 7% of waste in the mixtures to see if it made any difference.

DATE: 04/04/2023

TEXTILE:

TEXTILE SUBSTRATE	Polyester woven fabric
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COATING FORMULATION: 100g

	DESIGNATION	AMOUNT (g)	%	NOTES
POLYMER	POLYMER 4	94.3 92.3	94.3 92.3	PT8.1 PT8.2
WASTE	Pine Bark (previously dried and ball milled)	5 7	5 7	PT8.1 PT8.2
ADDITIVES: Thickener	THICKENER 1	q.b	-	-
Crosslinker	CROSSLINKER 2	0.7	0.7	-

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
scale	PG 5002 Deltarange
Mixer	Techmatic S2
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	-
DRYING	100 °C/ 5 min./ 1300 rpm*	Each layer dried separately
CURING	150 °C/ 5 min./ 1300 rpm*	After all layers applied
LAYERS	2x 0.0mm	-

*Fan Speed

OTHER INFO:

	VALUES	NOTES
VISCOSITY	8.99 Pa.s (8990 cps) 4.21 Pa.s (4210 cps)	100 rpm Torque: 89.9% s06* (8.1) 100 rpm Torque: 42.1% s06* (8.2)

* S means Spindle and the number is the identifier of the spindle.

NOTES:

- Order of mixing was polymer, then crosslinker mixed, then residue was added slowly, not all at the same time, while the paste was mixing continuously, then thickener was added drop by drop until visually it was less liquid, sticking to the spoon.
- The measurement of viscosity was made as soon as the paste was ready to be applied.
- These pastes were applied in a woven fabric made of a mixture of polyester and cotton.
- Two layers with 0.0mm were applied.
- In this test, previously dried and ball milled waste was used.

CONCLUSIONS/OBSERVATIONS/RESULTS:

- This test was made at the same time as #PT07 so in this one was also tested the usage of 7% of waste in the paste (PT08.2) as well as the use of 5% (PT08.1) since there weren't previous tests with the ball milled waste.
- None of the tests showed interesting results: the coating appears with many waste lumps which resulted in the appearance of many "strings" in the coating surfaces leading to a very uneven and very textured surface.
- The process of ball milling is proven to not be worth it to this project since it doesn't add any value, rather, the contrary.

EXPERIMENT #PT09.1 & #PT09.2

OBJECTIVE: To test the paste that uses 7% of pine bark waste with different amounts of layers and thickness.

DATE: 06/04/2023

TEXTILE:

TEXTILE SUBSTRATE	Polyester woven fabric
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COATING FORMULATION: 200g

	DESIGNATION	AMOUNT (g)	%
POLYMER	POLYMER 4	184.6	92.3
WASTE	Pine Bark (previously dried and ball milled)	7	7
ADDITIVES: Thickener	THICKENER 1	q.b	-
Crosslinker	CROSSLINKER 2	1.4	0.7

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
scale	PG 5002 Deltarange
Mixer	Techmatic S2
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Strainer	-
Calender Machine	Macpi 553.37-9124 FLAT BONDING PRESS
Calender Papers	SAPPI – U/C ADVA RAINBOW (947) 720A-178R SAPPI/WARREN RELEASE PAPERS - ULTRACAST UNIVERSAL HAMPTON 7060-204R SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	-
DRYING	100 °C/ 5 min./ 1300 rpm*	Each layer dried separately

CURING	150 °C/ 5 min./ 1300 rpm*	After all layers applied
LAYERS	2x 0.1mm 2x 0.0mm + 1x 0.1mm	#PT09.1 #PT09.2
CALENDERING	140 °C/ 30 seg. / 3 Bar	

*Fan Speed

OTHER INFO:

	VALUES	NOTES
VISCOSITY	9.54 Pa.s (9540 cps)	100 rpm Torque: 95.4% s06*

* S means Spindle and the number is the identifier of the spindle.

NOTES:

- Order of mixing was polymer, then crosslinker, then waste was added slowly, not all at the same time, while the paste was mixing continuously, then thickener was added drop by drop until visually it was less liquid, sticking to the spoon.
- The measurement of viscosity was made as soon as the paste was ready to be applied.
- After the viscosity measurement, the paste was strained, to remove and avoid lumps in the paste.
- This paste was applied in a woven fabric made of a mixture of polyester and cotton.
- In #PT09.1 two layers of 0.1mm were applied.
- In #PT09.2 two layers of 0.1mm were also applied with a last layer with 0.1mm
- The coating samples were embossed using the papers presented in the section "Materials Used" and calendered without any paper.

CONCLUSIONS/OBSERVATIONS/RESULTS:

- This test was made to test the layers necessary for the coating to look as desired regarding touch and colour.
- This paste turned out good which made us conclude that the viscosity values for this paste to work, and also having in account the other tests, must be between 5-10 Pa. s (5000-10000 cps).
- The fact that the paste was strained before application also helped significantly.
- The 3 layers work better than only 2.
- The calendaring was proven useful when using the paper SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R, whose purpose is to just smooth the surface, not having any special effects such as gloss treatments or textures. The surface indeed becomes smoother.
- The calendaring using no paper shows a smooth surface, however, the section in which the paper SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R was used is much smoother, proving it is a better option.
- The papers SAPPI – U/C ADVA RAINBOW (947) 720A-178R and SAPPI/WARREN RELEASE PAPERS - ULTRACAST UNIVERSAL HAMPTON 7060-204R were supposed to give effects of gloss treatments on the coating, however, the changes are not visible which means the best choice is indeed the use of SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R.

EXPERIMENT #PT10.1 & #PT10.2

OBJECTIVE: To test different types of thickeners.

DATE: 11/04/2023

TEXTILE:

TEXTILE SUBSTRATE	Polyester woven fabric
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COATING FORMULATION: 200g

	DESIGNATION	AMOUNT (g)	%	NOTES
POLYMER	POLYMER 4	184.6	92.3	-
WASTE	Pine Bark (previously dried and ball milled)	7	7	-
ADDITIVES: Thickener	THICKENER 1 THICKENER 2	q.b	-	10.1 10.2
Crosslinker	CROSSLINKER 2	1.4	0.7	-

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
scale	PG 5002 Deltarange
Mixer	Techmatic S2
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Strainer	-
Calender Machine	Macpi 553.37-9124 FLAT BONDING PRESS
Calender Papers	SAPPI – U/C ADVA RAINBOW (947) 720A-178R SAPPI/WARREN RELEASE PAPERS - ULTRACAST UNIVERSAL HAMPTON 7060-204R SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	-
DRYING	100 °C/ 5 min./ 1300 rpm*	Each layer dried separately
CURING	150 °C/ 5 min./ 1300 rpm*	After all layers applied

LAYERS	2x 0.0mm + 1x 0.1mm	
CALENDERING	140 °C/ 30 seg. / 3 Bar	

*Fan Speed

OTHER INFO:

	VALUES	NOTES
VISCOSITY	6.67 Pa.s (6670 cps) 6.68 Pa.s (6680 cps)	100 rpm Torque: 66.7% s06* (10.1) 100 rpm Torque: 66.8% s06* (10.2)

* S means Spindle and the number is the identifier of the spindle.

NOTES:

- Order of mixing was polymer, then crosslinker, then residue was added slowly, not all at the same time, while the paste was mixing continuously, then thickener was added drop by drop until visually it was less liquid, sticking to the spoon.
- The measurement of viscosity was made as soon as the paste was ready to be applied.
- These pastes were both strained 2 times before application to remove and avoid lumps in them.
- These pastes were applied in a woven fabric made of a mixture of polyester and cotton.
- Both samples have 2 layers with 0.0mm and one with 0.1mm.
- The coating samples were embossed using the papers presented in the section "Materials Used" and calendered without any paper.

CONCLUSIONS/OBSERVATIONS/RESULTS:

- This test was made to compare 2 different types of thickeners, the one that was being used (Thickener 17) and a different one (Thickener PUR 41) to test if the result with this last one made the paste smoother.
- The conclusions were that in fact, the result with Thickener 17, is better than with Thickener PUR 41, Thickener 17 gives the coating a smoother surface than Thickener PUR 41. This means the change is not considered useful.
- The improvements and conclusions from the experiments in #PTog tests were applied in this test and proven right.
- Also in these samples, the results in the calendaring were the same as presented in #PTog:
 - The calendaring was proven useful when using the paper SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R, which the purpose is to smooth the surface, not to provide any special effects such as gloss treatments or textures. The surface indeed becomes smoother.
 - The calendaring using no paper shows a smooth surface, however, the section in which the paper SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R was used is much smoother, proving it is a better option.
 - The papers SAPPI – U/C ADVA RAINBOW (947) 720A-178R and SAPPI/WARREN RELEASE PAPERS - ULTRACAST UNIVERSAL HAMPTON 7060-204R were supposed to give effects of gloss treatments on the coating, however, the changes are not visible which means the best choice is indeed the use of SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R.

EXPERIMENT #PT11.1 & #PT11.2

OBJECTIVE: Changes in crosslinker type and amount + number of layers and thickness testing.

DATE: 10/05/2023

TEXTILE:

TEXTILE SUBSTRATE	Polyester woven fabric
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COATING FORMULATION: 100g

	DESIGNATION	AMOUNT (g)	%	NOTES
POLYMER	POLYMER 4	92.65	92.65	-
WASTE	Pine Bark (previously dried)	7	7	-
ADDITIVES: Thickener	THICKENER 1	q.b	-	-
Crosslinker	CROSSLINKER 1	0.35	0.35	-
Topcoat	TOPCOAT 2	-	-	only on 11.1

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
scale	PG 5002 Deltarange
Mixer	Techmatic S2
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Strainer	-
Calender Machine	Macpi 553.37-9124 FLAT BONDING PRESS
Calender Papers	SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	-
DRYING	100 °C/ 5 min./ 1300 rpm*	Each layer dried separately
CURING	150 °C/ 5 min./ 1300 rpm*	After all layers applied
LAYERS	2x 0.0mm + 1x 0.1mm 1x 0.0mm + 2x 0.1 mm	11.1 11.2
CALENDERING	140 °C/ 30 seg./ 3 Bar	

*Fan Speed

OTHER INFO:

	VALUES	NOTES
VISCOSITY	7.07 Pa.s (7070 cps)	100 rpm Torque: 70.7% s06*

* S means Spindle and the number is the identifier of the spindle.

NOTES:

- Order of mixing was polymer, then crosslinker, then residue was added slowly, not all at the same time, while the paste was mixing continuously, then thickener was added drop by drop until visually it was less liquid, sticking to the spoon.
- The measurement of viscosity was made as soon as the paste was ready to be applied.
- This paste was strained 2 times before application to remove and avoid lumps in them.
- This paste was applied in a woven fabric made of a mixture of polyester and cotton.
- Sample #PT11.1 has 2 layers with 0.0mm and one layer with 0.1mm
- Sample #PT11.2 has 1 layer with 0.0mm and 2 layers with 0.1mm
- The sample 11.1 has been top coated and both samples have been calendared with the SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R paper.

CONCLUSIONS/OBSERVATIONS/RESULTS:

- This test was made because the paste had been behaving in a not normal way, it would dry a little bit while still in the cup yet to be applied. This meant that when applied, the paste was creating a lot of "strings" and lumps in the surface, although the paste was strained at least 2 times before application. So, to prevent this from happening the crosslinkers quantity was reduced to half of what, until 0,5% was being used (down from 0.7%).
- The result showed improvement as the paste stopped drying while still in the cup and it became easier to apply.
- Later, it was discovered that the initial information about the use of crosslinker was incorrect and that the value of 0.7% was indeed too high. It was discovered that the value in 100% should be 0.3% or lower.
- In the sample #PT11.1 it is visible yet, some strings even after the crosslinker change and that is why #PT11.2 was created in this test.
- The result shows indeed less "strings" which lead to believe that in fact the answer can be, in fact, in this change with the crosslinker combined with the layers thickness and arrangement.
- The sample #PT11.1 was top coated with a bio topcoat. Until this point a not bio topcoat was tested. The result shows a less sticky and smoother surface even without the calendaring done yet. The surface is less glossy and more matte.
- Both samples were calendared using the paper SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R. In the sample #PT11.1 there is a significant change in the surface texture since the embossing made the surface even smoother, where the particles of waste were less felt. The sample #PT11.2 also acquired a smoother surface, although, not as significant as in #PT11.1.

EXPERIMENT #PT12

OBJECTIVE: Number of layers and thickness testing. Decrease of the crosslinker amount to 0.3%.

DATE: 16/05/2023

TEXTILE:

TEXTILE SUBSTRATE	Polyester woven fabric
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COATING FORMULATION: 300g

	DESIGNATION	AMOUNT (g)	%
POLYMER	POLYMER 4	278.1	92.7
WASTE	Pine Bark (previously dried)	21	7
ADDITIVES: Thickener	THICKENER 1	q.b	-
Crosslinker	CROSSLINKER 1	0.9	0.3
Topcoat	TOPCOAT 2	-	-

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
scale	PG 5002 Deltarange
Mixer	Techmatic S2
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Strainer	-
Calender Machine	Macpi 553.37-9124 FLAT BONDING PRESS
Calender Papers	SAPPI - U/C UNIVERSAL CARRARA (971) 700W-176R
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	-
DRYING	100 °C/ 5 min./ 1300 rpm*	Each layer dried separately
CURING	150 °C/ 5 min./ 1300 rpm*	After all layers applied
LAYERS	2x 0.1mm	-
CALENDERING	140 °C/ 30 seg./ 3 Bar	

*Fan Speed

OTHER INFO:

	VALUES	NOTES
VISCOSITY	7.96 Pa.s (7960 cps)	100 rpm Torque: 79.6% s06*
PH	Not measured	-

* S means Spindle and the number is the identifier of the spindle.

NOTES:

- Order of mixing was polymer, then crosslinker, then residue was added slowly, not all at the same time, while the paste was mixing continuously, then thickener was added drop by drop until visually it was less liquid, sticking to the spoon.
- The crosslinker was decreased by 0.05% to its recommended amount.
- The measurement of viscosity was made as soon as the paste was ready to be applied.
- This paste was strained 2 times before application to remove and avoid lumps in it.
- The paste was applied in a woven fabric made of a mixture of polyester and cotton.
- The sample has 2 layers with 0.1mm each.
- The sample has been top coated and calendared with the SAPPI – U/C UNIVERSAL CARRARA (g71) 700W-176R paper.

CONCLUSIONS/OBSERVATIONS/RESULTS:

- This test was made to try and increase the height of the layers applied to the substrate. It is believed that the fact that the first layer was being applied with 0.0mm was one of the causes of the "stringy" appearance on the coating surface. So, the test was done to experiment applying layers with 0.1mm.
- The result shows, in fact, a much more homogeneous even surface.
- This is considered to be the last test with the pine bark waste, regarding this dissertation. This doesn't mean that this coating paste is completely ready to be industrialised. It is believed that it is very promising and can, in further studies, be improved to a more stable and homogeneous coating paste.

EXPERIMENT #PT13

OBJECTIVE: To test the use of pine sawdust waste.

DATE: 16/05/2023

TEXTILE:

TEXTILE SUBSTRATE	Polyester woven fabric
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COATING FORMULATION: 100g

	DESIGNATION	AMOUNT (g)	%
POLYMER	POLYMER 4	92.7	92.7
WASTE	Pine sawdust	7	7
ADDITIVES: Thickener	THICKENER 1	q.b	-
Crosslinker	CROSSLINKER 1	0.3	0.30

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
scale	PG 5002 Deltarange
Mixer	Techmatic S2
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Cups	-
Spoons	.

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	-
DRYING	100 °C/ 5 min./ 1300 rpm*	Each layer dried separately
CURING	150 °C/ 5 min./ 1300 rpm*	After all layers applied
LAYERS	3x 0.0mm	

*Fan Speed

OTHER INFO:

	VALUES	NOTES
VISCOSITY	6.35 Pa.s (6350 cps)	100 rpm Torque: 63.5% s06*

* S means Spindle and the number is the identifier of the spindle.

NOTES:

- Order of mixing was polymer, then crosslinker, then waste was added slowly, not all at the same time, while the paste was mixing continuously, then thickener was added drop by drop until visually it was less liquid, sticking to the spoon.
- The measurement of viscosity was made as soon as the paste was ready to be applied.
- The paste was applied in a woven fabric made of polyester.
- The sample has 3 layers with 0.0mm each.

CONCLUSIONS/OBSERVATIONS/RESULTS:

- This test was made using the pine sawdust that came from Chile.
- Since the paste using pine bark had been tested before, this test was done using the same formulation products and amounts to test if it behaved equally well.
- The result shows, in fact, that it behaves very well, showing no lumps or "strings". It presents an even and homogeneous surface.
- It is concluded that the sawdust behaves better when applied than the bark.
- The result shows a very light-coloured surface. It is desired a darker shade.

EXPERIMENT #PT14.1 and #PT14.2

OBJECTIVES: Test the use of 10% amount of waste in the formulation and layers amount and thickness.

DATE: 22/05/2023

TEXTILE:

TEXTILE SUBSTRATE	Polyester woven fabric
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COATING FORMULATION: 100g

	DESIGNATION	AMOUNT (g)	%
POLYMER	POLYMER 4	89.7	89.7
WASTE	Pine sawdust	10	10
ADDITIVES: Thickener	THICKENER 1	q.b	-
Crosslinker	CROSSLINKER 1	0.3	0.3
Topcoat	TOPCOAT 2	-	-

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
scale	PG 5002 Deltarange
Mixer	Techmatic S2
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Calender Machine	Macpi 553.37-9124 FLAT BONDING PRESS
Calender Papers	SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	-
DRYING	100 °C/ 5 min./ 1300 rpm*	Each layer dried separately
CURING	150 °C/ 5 min./ 1300 rpm*	After all layers applied
LAYERS	3x 0.0mm 3x 0.1mm	14.1 14.2
CALENDERING	140 °C/ 30 seg./ 3 Bar	

*Fan Speed

OTHER INFO:

	VALUES	NOTES
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VISCOSITY	3.93 Pa.s (3930 cps)	100 rpm Torque: 39.3% s06*
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* S means Spindle and the number is the identifier of the spindle.

NOTES:

- Order of mixing was polymer, then crosslinker, then residue was added slowly, not all at the same time, while the paste was mixing continuously, then thickener was added drop by drop until visually it was less liquid, sticking to the spoon.
- The measurement of viscosity was made as soon as the paste was ready to be applied.
- The paste was applied in a woven fabric made of polyester.
- This test presents 2 different samples, 14.1 and 14.2, both using the same paste. #PT14.1 has 3 layers with 0.0mm each and #PT14.2 has 3 layers with 0.1mm each.
- In part of the samples, a topcoat was applied to test how it looked and felt in this coating as well as part was also embossed using the paper SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R.

CONCLUSIONS/OBSERVATIONS/RESULTS:

- This test was done to try increasing the % of waste in the paste from 7% (the amount used in the first test with the sawdust) to 10%. The increase did not create problems, in fact it delivers a more homogeneous surface.
- The surface is somewhat less smooth than the sample from #PT13.
- #PT14.1 has the same number and layers and height as #PT13. There is a slight change in colour although it is not significant. Which means the colour intensity doesn't come from the increase of percentage of waste in the formulation.
- #PT14.2 has thicker layers, and the results are visible. It acquired a darker shade, and the surface looks more homogeneous. Also, the touch feels smoother.
- A topcoat was applied in a part of both samples: in both the surface becomes smoother and matter (less glossy) as well as less sticky. In #PT14.1 it is still possible to feel some rough surface but in #PT14.2 it acquires a very smooth surface.
- Both samples were embossed, in the part where topcoat was applied as well as in the part that doesn't have topcoat. In the part that was topcoated the embossing made the surface softer for both samples. The same happened in the part that didn't have topcoat but the difference between this part, embossed or not embossed, it's not as prominent as the difference in the part that has topcoat.

EXPERIMENT #PT15

OBJECTIVE: Test obtaining a darker shade surface by trying a different amount and thickness of the layers.

DATE: 22/05/2023

TEXTILE:

TEXTILE SUBSTRATE	Polyester woven fabric
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COATING FORMULATION: 100g

	DESIGNATION	AMOUNT (g)	%
POLYMER	POLYMER 4	89.7	89.7
WASTE	Pine sawdust	10	10
ADDITIVES: Thickener	THICKENER 1	q.b	-
Crosslinker	CROSSLINKER 1	0.3	0.3
Topcoat	TOPCOAT 2	-	-

TOOLS:

DESIGNATION	BRAND & SPECIFICATIONS
scale	PG 5002 Deltarange
Mixer	Techmatic S2
Application Machine	Mathis AG LABCOATER type «LTE-S»
Viscometer	Brookfield DVE Viscometer
Calender Machine	Macpi 553.37-9124 FLAT BONDING PRESS
Calender Papers	SAPPI – U/C UNIVERSAL CARRARA (971) 700W-176R
Cups	-
Spoons	-

PROCESS OF APPLICATION:

	VALUES	NOTES
TECHNIQUE	Knife on roll	-
DRYING	100 °C/ 5 min./ 1300 rpm*	Each layer dried separately
CURING	150 °C/ 5 min./ 1300 rpm*	After all layers applied
LAYERS	1x 0.1mm + 1x 0.2mm + 1x 0.3mm + 1 x 0.4mm	
CALENDERING	140 °C/ 30 seg./ 3 Bar	

*Fan Speed

OTHER INFO:

	VALUES	NOTES
VISCOSITY	3.93 Pa.s (3930 cps)	100 rpm Torque: 39.3% s06*

* S means Spindle and the number is the identifier of the spindle.

NOTES:

- Order of mixing was polymer, then crosslinker, then residue was added slowly, not all at the same time, while the paste was mixing continuously, then thickener was added drop by drop until visually it was less liquid, sticking to the spoon.
- The measurement of viscosity was made as soon as the paste was ready to be applied.
- The paste was applied in a woven fabric made of a mixture of polyester and cotton.
- This test presents a sample that contains 4 layers: 1 with 0.1mm one with 0.2mm; one with 0.3mm and one with 0.4mm.
- In part of the sample, a topcoat was applied to test how it looked and felt in this coating and part of the sample was also embossed using the paper SAPPI – U/C UNIVERSAL CARRARA (g71) 700W-176R.

CONCLUSIONS/OBSERVATIONS/RESULTS:

- The results are considered good. Although, it presents some strings that are quite imperceptible but are there.
- The shade is darker but the difference to #PT14.2 is not relevant.
- Topcoat was applied in half of this sample and the result is very similar, if not the same, to the test #PT14.2.
- Both samples were embossed, in the part where topcoat was applied as well as in the part that doesn't have topcoat. In the part that was topcoated the embossing made the surface softer for both samples. The same happened in the part that didn't have a topcoat but the difference between this part, embossed or not embossed, it's not as prominent as the difference in the part that has a topcoat.
- It was concluded that, #PT14.2 showed a more even surface and a better result than this test. Although this test presents a darker shade, the difference is not relevant enough to choose this coating method.

DEPARTAMENTO DE LABORATÓRIOS



À Firma

CENTRO TEC. IND. TEX. E DO VES. DE PORTUGAL (SEDE)
RUA FERNANDO MESQUITA, Nº 2785

4760-034- VILA NOVA FAMALICAO

Entrada: 1429/2023

Data de Recepção das Amostras : 2023/06/22

Observações

Este relatório tem aditamento

N. Amostras - V/Referência

3940/2023 - Amostras de "Serrim"

3941/2023 - Amostras de "Casca de pinheiro"

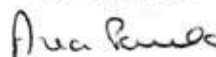
Ensaio(s) Requeridos

Projecto BE&T - 15

- Os ensaios foram realizados entre a data 2023/06/22 e 2023/06/30.

V.N.FAMALICÃO, 30 de Junho de 2023

COORDENADOR
DO LABORATÓRIO



(Eng^a Ana Paula Fonte)

NOTAS:

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l.q.-limite de quantificação, l.d.-limite de deteção e n.d.-não detetado.

DEPARTAMENTO DE LABORATÓRIOS

No. da Amostra	V/ Referência	Descrição da Amostra
3940 /2023	Amostras de "Serrim"	3 Amostras de material

Ensaio/Norma: * RESISTÊNCIA À ABRASÃO (MARTINDALE) / ISO 5470-2

Resultados

Grau de degradação	
Valores individuais	
1600 revoluções	0 0
3200 revoluções	0 0
6400 revoluções	0 0
12800 revoluções	0 0
25600 revoluções	0 0
38400 revoluções	1 1
51200 revoluções	1 1

Condições de Ensaio

Lado testado -	Direito
Número de provetes testados -	2 (por escassez de amostra)
Tipo de abrasímetro - Martindale	
Tipo de abrasivo - Tecido de lã	
Pressão aplicada (KPa)-	12
Método usado-	1
Ambiente acondicionado:	
20±2°C e 65±4% H.R.	
Classificação de 0 a 5 em que 0	
é sem alteração e 5 é degradação	
completa.	

Ensaio/Norma: SOLIDEZ DO TINTO À FRICÇÃO A SECO / ISO 105 X12:2016

Resultados

Manchamento (teia ou comp/trama ou larg)	4-5
--	-----

Condições de Ensaio

Nota: o valor indicado corresponde ao
pior valor encontrado.
Dedo de fricção usado: redondo
Força aplicada: 9 N
Direcção testada: teia e trama ou
comprimento e largura
Condições de condicionamento: min. 4 h a
20+/-2°C e 65+/-4% H.R.

Ensaio/Norma: SOLIDEZ DO TINTO À FRICÇÃO A HÚMIDO / ISO 105 X12:2016

DEPARTAMENTO DE LABORATÓRIOS

Resultados

Manchamento (teia ou comp/trama ou larg) 4

Condições de Ensaio

Nota: o valor indicado corresponde ao
pior valor encontrado.
Dedo de fricção usado: redondo
Força aplicada: 9 N
Direção testada: teia e trama ou
comprimento e largura
Percentagem de molhagem: 95% a 100%
Condições de condicionamento: min 4 h a
20+/-2°C e 65+/-4% H.R.

No. da Amostra	V/ Referência	Descrição da Amostra
3941 /2023	Amostras de "Casca de pinheiro"	3 Amostras de material

Ensaio/Norma: * RESISTÊNCIA À ABRASÃO (MARTINDALE) / ISO 5470-2

Resultados

Grau de degradação	0	0	0	0
Valores individuais	0	0	0	0
1600 revoluções	0	0	0	0
3200 revoluções	0	0	0	0
6400 revoluções	0	0	0	0
12800 revoluções	1	1	1	1
25600 revoluções	1	1	1	1
38400 revoluções	2	2	2	2
51200 revoluções	2	2	2	2

Condições de Ensaio

Lado testado -	Direito
Tipo de abrasímetro - Martindale	
Tipo de abrasivo - Tecido de lã	
Pressão aplicada (KPa)-	12
Método usado-	1
Ambiente acondicionado:	
20±2°C e 65±4% H.R.	
Classificação de 0 a 5 em que 0	

DEPARTAMENTO DE LABORATÓRIOS

é sem alteração e 5 é degradação completa.

Ensaio/Norma: SOLIDEZ DO TINTO À FRICÇÃO A SECO / ISO 105 X12:2016

Resultados

Manchamento (teia ou comp/trama ou larg)	4-5
--	-----

Condições de Ensaio

Nota: o valor indicado corresponde ao pior valor encontrado.
Dedo de fricção usado: redondo
Força aplicada: 9 N
Direcção testada: teia e trama ou comprimento e largura
Condições de condicionamento: min. 4 h a 20+/-2°C e 65+/-4% H.R.

Ensaio/Norma: SOLIDEZ DO TINTO À FRICÇÃO A ÚMIDO / ISO 105 X12:2016

Resultados

Manchamento (teia ou comp/trama ou larg)	4-5
--	-----

Condições de Ensaio

Nota: o valor indicado corresponde ao pior valor encontrado.
Dedo de fricção usado: redondo
Força aplicada: 9 N
Direcção testada: teia e trama ou comprimento e largura
Percentagem de molhagem: 95% a 100%
Condições de condicionamento: min 4 h a 20+/-2°C e 65+/-4% H.R.

DEPARTAMENTO DE LABORATÓRIOS



À Firma

CENTRO TEC. IND. TEX. E DO VES. DE PORTUGAL (SEDE)
RUA FERNANDO MESQUITA, Nº 2785

4760-034- VILA NOVA FAMALICAO

Entrada: 1429/2023

Data de Recepção das Amostras : 2023/06/22

Observações

Este Relatório é aditamento do Relatório N.1504/2023-1

N. Amostras - V/Referência

3940/2023 - Amostras de "Serrim"

3941/2023 - Amostras de "Casca de pinheiro"

Ensaio(s) Requerido(s)

Projecto BE@T - 15

- Os ensaios foram realizados entre a data 2023/06/22 e 2023/07/03.

V.N.FAMALICÃO, 04 de Julho de 2023

COORDENADOR
DO LABORATÓRIO



(Engª Ana Paula Fonte)

NOTAS:

Os resultados deste relatório referem-se apenas às amostras submetidas a ensaio, não sendo da responsabilidade do laboratório a sua amostragem. O relatório de ensaios não pode ser parcialmente reproduzido sem autorização do CITEVE. Os ensaios assinalados com * não estão incluídos no âmbito da acreditação do laboratório e os ensaios assinalados com ** estão fora do âmbito da acreditação devido a alteração do método. Todos os serviços prestados estão de acordo com o documento "Termos & Condições do Serviço CITEVE" disponível em www.citeve.pt (<http://www.citeve.pt>). Na declaração de conformidade, a regra de decisão não tem em consideração a incerteza associada ao ensaio, com exceção dos casos em que no texto da conformidade está definida uma regra de decisão diferente (ver o documento "Política de regras de decisão na avaliação da conformidade dos Laboratórios CITEVE" publicada em www.citeve.pt (<http://www.citeve.pt>)). A incerteza expandida apresentada foi calculada multiplicando a incerteza padrão pelo fator de expansão k=2, que para uma distribuição normal permite associar ao resultado um nível de confiança de aproximadamente 95%.
I.q.-limite de quantificação, I.d.-limite de deteção e n.d.-não detetado.

DEPARTAMENTO DE LABORATÓRIOS

<u>No. da Amostra</u>	<u>V/ Referência</u>	<u>Descrição da Amostra</u>
3940 /2023	Amostras de "Serrim"	3 Amostras de material

Ensaio/Norma: SOLIDEZ DO TINTO À LUZ ARTIFICIAL / ISO 105-B02:2014

Resultados

Grau de solidez à luz: 4-5

Condições de Ensaio

Aparelho usado: Lâmpada de xénon
arrefecida por ar
Método utilizado: 3 (BS-5)
Temp. termómetro negro padrão: 47+/-3°C
Teste de humidade: grau 5
Avaliação com escala de azuis

<u>No. da Amostra</u>	<u>V/ Referência</u>	<u>Descrição da Amostra</u>
3941 /2023	Amostras de "Casca de pinheiro"	3 Amostras de material

Ensaio/Norma: SOLIDEZ DO TINTO À LUZ ARTIFICIAL / ISO 105-B02:2014

Resultados

Grau de solidez à luz: 3-4

Condições de Ensaio

Aparelho usado: Lâmpada de xénon
arrefecida por ar
Método utilizado: 3 (BS-5)
Temp. termómetro negro padrão: 47+/-3°C
Teste de humidade: grau 5
Avaliação com escala de azuis

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Environmental Footprints and Eco-design of Products and Processes

Biomimetics, Biodesign and Bionics

Technological Advances Towards a Sustainable Development

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About the Project

Nature is a vast source of inspiration and information for the resolution of complex problems, based on billions of years of natural selection, in terms of symbiosis, efficiency, and equilibrium between multiple forms of life. By means the analysis, organization, and modeling of biological features – such as patterns, shapes, mechanisms, colors, structures, and more – they can be transcribed for application in human creations. Therefore, humanly-designed projects can benefit from solutions that were already evaluated and verified throughout a harsh evolutionary environment. As a result, such outstanding characteristics can be sought after for new insights into the needs of our development.

With the advancements provided by new technologies for the investigation, simulation, and testing of natural features, the transcription process between nature and product can be accelerated. In this regard, this book project aims to highlight some of the recent technological breakthroughs in Biomimetics, Biodesign, and Bionics that contribute to the development of sustainable and innovative projects.

Scope of the Book

Biomimetics, Biodesign and Bionics are some of the most exciting fields of applied sciences due to their inherent innovation and synergy with sustainable development. As a result, a large interest has been driven by different disciplines – apart from Engineering, Architecture, and Design, due to their practical applications – such as Biology, Physics, Chemistry, and Mathematics. On the other hand, as new technological advances are being discovered, so is our crescent comprehension of natural characteristics as well as the ways we can apply them.

This title welcomes high-quality contributions regarding all aspects regarding Biomimetics, Biodesign, and Bionics (Bio*) and the recent technologies associated with sustainable applications. Areas that cover the biology-project research and development under the Sustainability umbrella will be considered for this book. Some of the areas of interest are mainly focused on – **but not limited to** – the fields of:

- Parametric Design
- New Materials and Processes
- Industrial Applications
- Technologies for Bio*
- Nature-Driven Innovations
- Bio* for Sustainable Development

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We will be receiving **Original** or **Review Chapters** about the above-mentioned topics. You may reply to us with details of your contribution, attached as a Word/PDF:

- Intended Chapter Title;
- Abstract (150 words);
- List of author names and affiliations;

Abstracts need to be submitted with the above details on or before **July 21, 2023**. The authors of selected Abstracts would be notified by **July 25 2023**, and will be asked to submit their full chapter for publication by **September 25 2023** (2 months to develop a chapter). The final book is tabled to be published by **November 2023**.

Authors of published chapters will be entitled to a free electronic copy of the final book. The title will be published in the “*Environmental Footprints and Eco-design of Products and Processes*” book series from Springer-Nature.

There are **NO COSTS/FEES/CHARGES** for the contributing authors throughout the project.

