Optimized smart manufacturing of technical textiles, their characterization and validation in relevant environment

Deliverable 4.3

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List of Abbreviations

Acronyms	Description
BS	British Standard.
CO	Cotton
conc.	Concentration
D	Deliverable
deg	Degree
EDS	Energy Dispersive Spectroscopy
FTIR	Fourier Transform Infrared Spectroscopy
GA	Grant agreement
ISO	International Organization for Standardization
P3HB	Poly(3-hydroxybutyrate)
PES	Polyester
PHA	Polyhydroxyalkanoate
PHB.E.0	Poly(3-hydroxybutyrate) (PH3B) diol polymer
PLA	Polylactic acid
S.D.	Standard deviation
SEM	Scanning Electron Microscopy
WP	Work Package

1. Introduction

The work here presented focused on evaluating the feasibility of scaling up the lab formulated polyhydroxyalkanoate (PHA) dispersion for fabric (spray) coating applications, and their characterization. To achieve this objective the work was divided into the following sub-tasks:

- **Production of a stable PHA dispersion suitable to be processed by spray**

Efforts focused on further improving the PHA dispersion developed in Task 3.3, namely by identifying an optimal PHA concentration and polymeric particle size to ensure the viability of the spray process. Additionally, appropriate concentrations of surfactants and dispersants were further investigated to enhance dispersion stability. Parameters such as sonication time and stirring duration were also optimized to achieve homogeneous and stable dispersions.

After achieving the most stable dispersion, this was up scaled for its application at an industrial setting.

- **Production of PHA spray-coated textile substrates**

The spray technique was employed to develop this type of coatings. The application of PHA dispersions onto textile substrates via spray was further investigated to determine the most suitable processing parameters for effective coating performance. Various factors were studied to optimize the coating process, with the aim of ensuring uniform deposition and strong adhesion. Cotton and polyester fabrics were used as substrates to evaluate the properties imparted by the PHA coatings.

- **Characterization of PHA coated textile substrates**

All PHA-coated textile substrates were subjected to various characterization tests to confirm the presence of PHA on the substrates and to evaluate the properties imparted by the coating.

- **Development of the sportswear demonstrator**

After production of the PHA-coated textiles, these were used to develop two padel outfits, as demonstrators of the sportswear application of these coated textiles.

This report details the development of the textile demonstrators and their characterization. This is part of WP4 – Use cases and validation of bio-based products and processes.

This report, together with D4.1, D4.2 and D4.4, demonstrates the accomplishment of MS5 (Bio-based demonstrators for each VC), by showing the production of spray coated textiles and their validation in relevant environments (simulated usage conditions) displaying promising properties, although requiring improvement.

2. Optimized smart manufacturing of technical textiles, their characterization and validation in relevant environment

This section provides a detailed account of the activities and progress made under Task 4.3. Each sub-task was addressed systematically to meet the overall objectives of the task.

2.1. Production of a stable PHA dispersion suitable to be processed by spray

To develop a PHA dispersion suitable for spray application, several steps were undertaken under T3.3 (and described in Deliverable 3.3).

As described in D3.3., from the PHAs produced in the project, the one most promising for spray coating was PHB.E.0, a Poly(3-hydroxybutyrate) (PH3B) diol polymer, due to its low molecular weight and improved dispersibility in aqueous media. Its dispersion in water was achieved with the help of a surfactant and a dispersant, for which the compositions and concentrations were tested to achieve the best result. Also, the granulometry of the polymer was studied and as expected, the lower the granulometry, the most uniform was the dispersion obtained. Furthermore, the stirring and sonication time were also accessed.

The optimized aqueous dispersion was prepared by dispersing 0.5–4% w/v of PHB.E.0 (particle size ≤ 60 µm) in distilled water under continuous stirring for 30 minutes. To ensure the effective activation of the surfactant and dispersant, the pH of the solution was adjusted to neutral. Next, 0.5–6% w/v of surfactant and 0.5–3% w/v of dispersant were added to the solution, followed by stirring for 1 hour. To achieve a homogeneous and stable dispersion, the mixture was then subjected to ultrasonic treatment.

2.2. Production of PHA spray-coated textile substrates

Once the optimized PHA dispersion was produced, the coating process was carried out first in lab-scale, to confirm the feasibility of this dispersion. Parameters such as spraying time, pressure, and nozzle type were varied.

In short, the prepared PHA dispersion was applied by spray to polyester (PES) and cotton (CO) fabric samples using an AutoJet® E1850+ spray system equipped with a hydraulic nozzle. The spray parameters were set as follows: 100% flow rate, 5 bar solution pressure, and 10 seconds of application per sample.

After deposition, the coated fabrics were dried at 100 °C for 10 minutes, followed by thermofixation at 150 °C for 5 minutes. To assess whether heat-assisted pressing could improve polymer adhesion to the textile substrates, a hot-press process was performed on selected samples at 180 °C for 15 seconds under 5 bars, aiming to enhance the fixation efficiency. All the process is shown below in Figure 1.

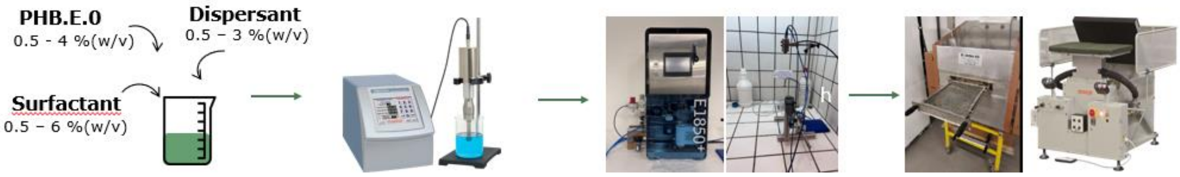

Figure 1 Development process of PHA spray-coated textile substrates.

2.3. Characterization of PHA coated textile substrates

Various characterization tests were conducted to analyse the presence of PHA (Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR), Energy Dispersive Spectroscopy (EDS)) on the substrates and to evaluate its influence on the properties of the resulting coated textiles (wettability, water absorption and air and vapor permeabilities).

To analyse the presence of PHA on the substrates, morphological analyses of the fabrics coated with PHB.E.0 dispersions were conducted using SEM (Figure 2). This showed that PHA particles are clearly present on both PES and CO substrates. These SEM images demonstrate the effectiveness of the spray coating process, confirming successful deposition of the polymer onto the textile surfaces.

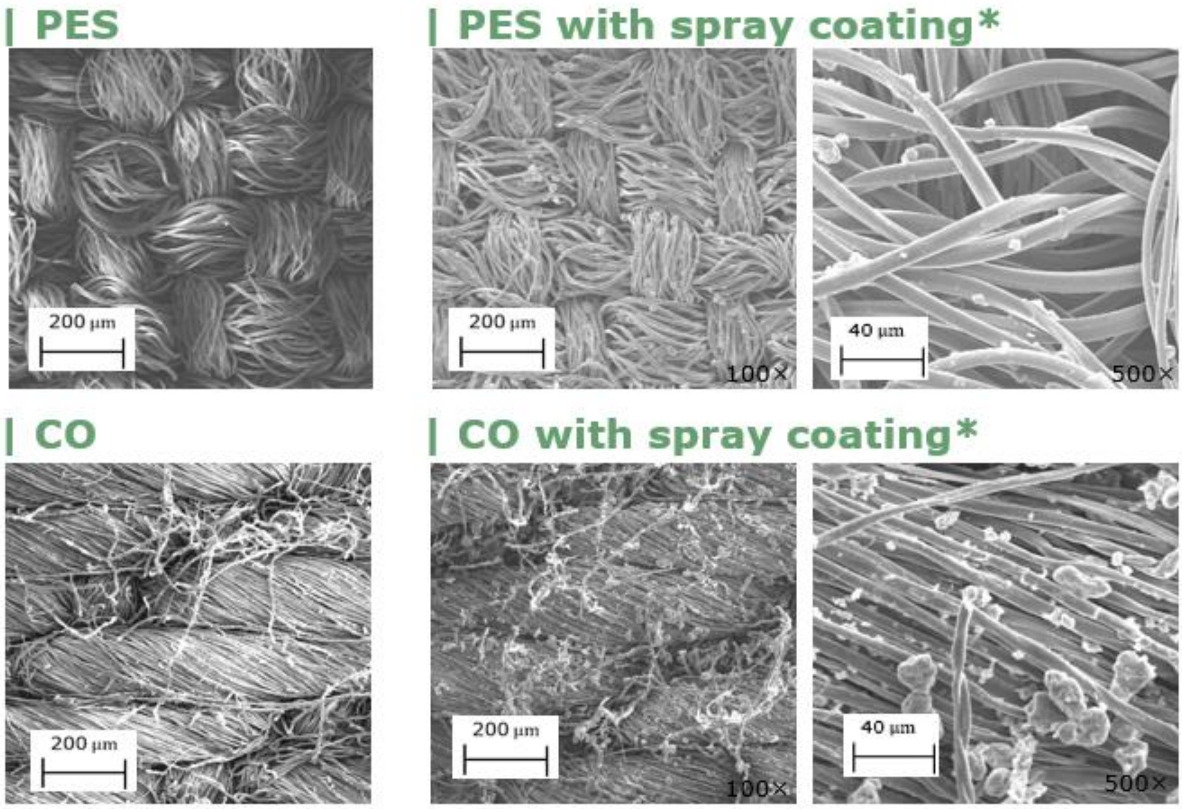

Figure 2 SEM micrographs of PHA-coated polyester and cotton control fabrics. The left images represent the control and those at right presented the coated substrates.

Subsequently, FTIR analyses were performed to further confirm the presence of the PHA. On the FTIR spectrum of PES samples (Figure 3, left), a peak at 1723 cm^{-1}, corresponding to the C=O (carbonyl) stretching of the ester groups in PHA, is clearly observed. This peak exhibits higher intensity in the coated samples compared to the control, indicating successful incorporation of the polymer. Additionally, a distinct peak at 1455 cm^{-1}, associated with the asymmetric bending (scissoring) of $-CH_2-$ and $-CH_3$ groups, is observed only in the PHA-coated PES samples.

In the case of the CO samples, the FTIR spectra (Figure 3, right) also shows slight differences between the coated and control fabrics. These subtle variations are likely due to the lower amount of PHB.E.0 used in the coating process. Nevertheless, the characteristic peaks at 1723 cm^{-1} and 1455 cm^{-1} are still present in the coated samples. Furthermore, a peak at 1376 cm^{-1}, corresponding to the symmetric bending (or wagging) of –CH$_3$ groups, is also clearly identified in the PHA-coated CO fabrics, further confirming the presence of the polymer.[1]

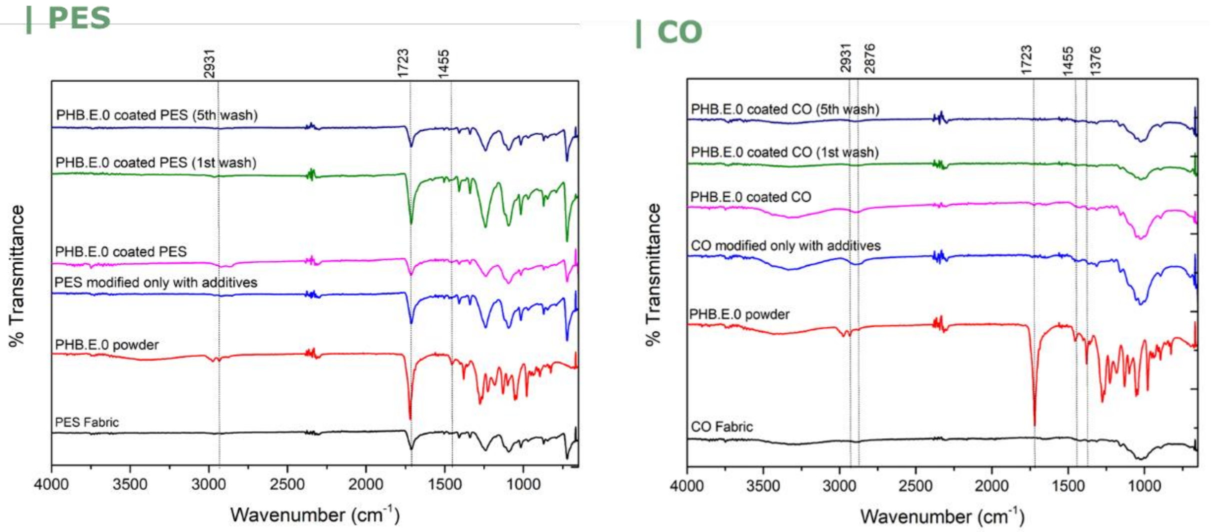

Figure 3 FTIR spectra of modified PES and CO fabrics.

In addition to FTIR, EDS was performed to further confirm the presence of PHA on the textile substrates. Carbon and oxygen were the key elements analysed, as they are the primary constituents of the PHA polymer chain. As shown in Table 1 and Table 2, PHA-coated samples exhibited higher atomic concentrations of carbon and oxygen compared to the uncoated controls, mainly for PES. These results provide additional evidence of successful deposition of the PHB.E.0 coating onto the fabric surfaces.

Table 1 EDS results of PHA coated PES substrates

Element name	Modification	Atomic conc. (%)	Weight conc. (%)
Carbon	Control	59.79±5.12	49.72± 5.32
	PHB.E.0 coated	61.02±3.11	53.41±3.08
	PHB.E.0 coated (hot-press)	60.42±2.69	52.76±2.71
Oxygen	Control	35.74±7.01	39.73±8.74
	PHB.E.0 coated	38.65±3.23	45.04±3.62
	PHB.E.0 coated (hot-press)	38.34±3.37	44.54±3.70

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[1] https://doi.org/10.1007/s10924-021-02121-6

Table 2 EDS results of PHA coated CO substrates

Element name	Modification	Atomic conc. (%)	Weight conc. (%)
Carbon	Control	48.27±8.43	41.40±8.54
	PHB.E.0 coated	46.81±1.66	36.68±6.11
	PHB.E.0 coated (hot-press)	54.69±9.37	47.79±9.53
Oxygen	Control	51.73±8.43	58.60±8.54
	PHB.E.0 coated	48.52±11.19	52.96±16.19
	PHB.E.0 coated (hot-press)	45.31±9.37	52.21± 9.53

Regarding the hydrophilicity of the samples, all the PHA-coated substrates showed to be hydrophilic (as shown in Table 3).

Table 3 Contact angles of different substrates

Fabric		Sample	Contact Angle (deg) ± S.D.[2]
PES	Control	Original fabric	63.93 ± 18.01
		Hot Pressing (160 °C)	50.30 ± 1.20
		Hot Pressing (180 °C)	86.96 ± 2.96
		Coated with additives*	Absorbs instantly
	PHB.E.0 coating	Without hot pressing	Absorbs instantly
		Hot Pressing (160 °C)	Absorbs instantly
		Hot Pressing (180 °C)	49.73 ± 7.87
CO	Control	Original fabric	Absorbs instantly
		Coated with additives*	
	PHB.E.0 coating	Without hot pressing	
		Hot Pressing (160 °C)	
		Hot Pressing (180 °C)	

* Surfactant and dispersant

To complement the contact angle measurements, a water absorption test was also conducted, according to ISO 20158:2018. This analysis provides insight into the amount of water absorbed by each substrate, both with and without PHA coating.

As shown in Figure 4, PES fabrics exhibited higher water absorption than CO, suggesting that the polyester samples had undergone specific treatments that increased their hydrophilicity, although at a slower ratio, given the contact angle values (Table 3). However, the data also indicates that the PHA coating had little to no effect on water absorption, as no significant differences were observed

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[2] Performed via the sessile drop measuring method (droplets of 3 µL of water, behaviour analysed for up to 90 s). At least, 5 measurements were done for each sample.

between coated and uncoated samples. This limited effect may be associated with the low concentration of PHA applied to the fabric surfaces, which may not have been sufficient to significantly alter their water absorption behaviour. On the other hand, the hot-press process did had effect on the water absorption capacity of the PES fabric (increasing by 7% at 160 °C and by 15% at 180 °C), probably due to changes in the fabric weave.

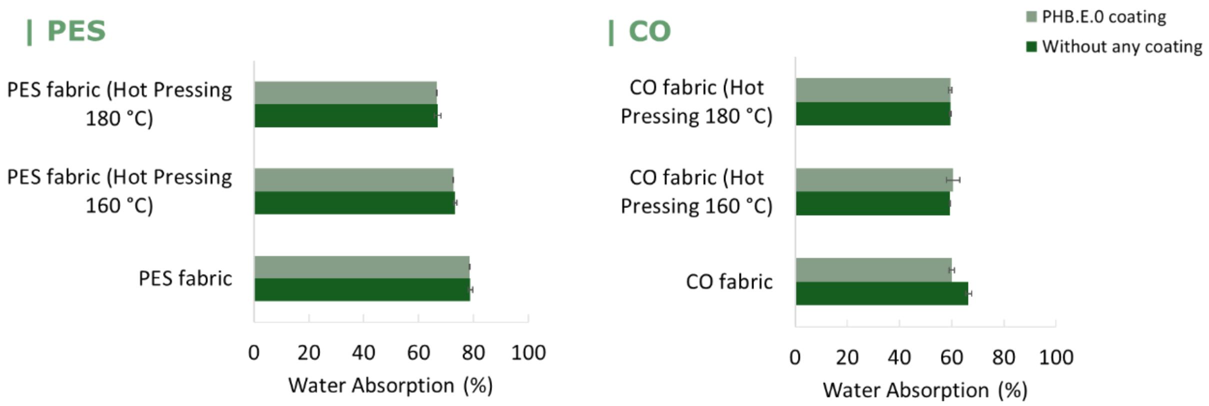

Figure 4 Water absorption of PES and CO fabrics: control samples and PHA-coated samples (with and without hot pressing).

In addition, air, and water vapor permeabilities were also analysed, according to ISO 9237:1997 and BS 7209:1990, respectively.

As seen in Figure 5, the air permeability decreases when the PES substrate is coated with PHA, compared to the uncoated substrate. This reduction may be advantageous, as it could enhance the material's resistance to external air penetration, providing better protection for the user's skin, conferring increased wind protection. A similar, though less pronounced, effect was also observed for the CO substrate. For all samples the air permeability of CO is significantly lower than that of PES, both before and after coating, which is expected given the differences on the fabrics densities (CO 369 g/m^2, PES 235 g/m^2).

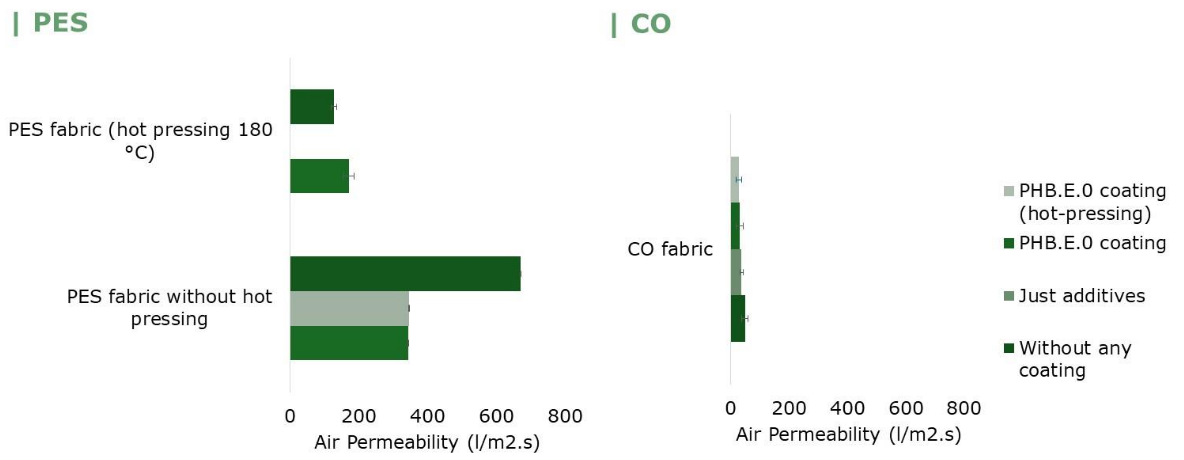

Figure 5 Air permeabilities of the PES and CO substrates.

Regarding water vapor permeability (Figure 6), no significant change was observed following the application of the coating on either PES or CO substrates. This indicates that the coating does not interferes with the fabric's moisture management properties.

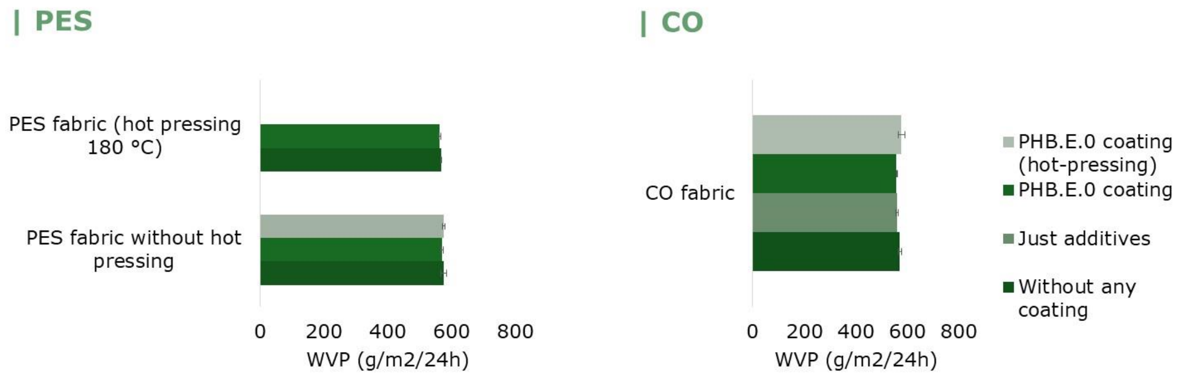

Figure 6 Water vapor permeabilities of the PES and CO substrate.

Therefore, these analyses indicate that further investigations and developments are needed. However, the results provide promising evidence that these coatings could enhance the performance of textiles, particularly during activities such as sports.

2.4. Scale-up

After the application of the improved dispersion in lab scale, an attempt was made to scale-up this dispersion, to be able to apply it in an industrial set-up at partner RIOPELE. The main goal of the scale-up attempt was to evaluate the feasibility of applying the lab-formulated PHA dispersion in fabric coating processes on a larger scale.

The spray system at RIOPELE is a WEKO-neo finishing equipment that works on a minimum amount of 30 L. Given the reactor available, 40 L of dispersion were prepared. As shown in Table 4, several parameters needed adjustment, namely the mixing methods. A mechanical overhead stirring and a sonicator were applied, as shown in Figure 7.

Table 4 Parameters used on lab and pilot scale

Parameter	Lab scale	Pilot Scale
Batch Volume	100 mL	40 L
Mixing Method	Magnetic stirring + Sonicator	Mechanical overhead stirring + Sonicator
Spray Equipment	AutoJet® E1850+ Spray System	WEKO-neo finishing

Figure 7 Mechanical overhead stirring and sonicator used in the scale-up phase.

Unfortunately, the scale-up did not give the intended result. Although the mixing time was increased to account for the scale-up (from 100 mL to 40 L), the obtained dispersion was not uniform, and some agglomerated particles were observed. Even after a filtration step, the aggregates formed again, and when an attempt was done to feed the WEKO spray system with this dispersion, the amount and size of aggregates was too big, and the filter at the entrance of the equipment clogged (Figure 8). As a result, the application was deemed unfeasible to continue, and due to timeline restraints, no other scale-up attempt could be carried out.

Figure 8 Clogging issues encountered during the spray coating process.

This problem was likely due to insufficient agitation and sonication times at the industrial scale, which compromised the stability of the dispersion. The preparation of 40 L of PHA dispersion appears to require longer processing than the 4 hours applied, even with the use of appropriate mechanical overhead stirring and sonication equipment.

Therefore, some adjusts are recommended to achieve a successful spray coating that includes an increase dispersion and sonication times, as well as improvement of the polymer particle size used. Implementing these improvements is expected to enhance the stability and performance of the PHA dispersion, enabling its effective application in large-scale spray coating processes.

2.5. Development of the sportswear demonstrator

2.5.1. Textiles selection

A user-centred, life-cycle-aligned material selection strategy was implemented, with a focus on environmental impact reduction and end-of-life value recovery. This includes:

- **Monomaterial strategy:** using a single material—recycled polyester (filament)—simplifies the recycling process at the garment's end of life, improving the product's overall circularity;
- **Recycled and recyclable inputs:** the fabric is derived from recycled plastic sources and is itself recyclable, demonstrating a high circularity potential. Compared to conventional spun polyester, filament polyester significantly reduces microplastic release;
- **Alternative bioplastic consideration:** polylactic acid (PLA) was also evaluated as a comparative sustainable material. While PLA offers the advantages of being biodegradable and derived from renewable sources, PES was selected for its superior performance in elasticity and durability for high-intensity sports applications.

Table 5 Material properties overview

Properties	Recycled Polyester (filament)	PLA (bioplastic)
Source	Recycled plastics	Renewable (corn, etc.)
End-of-life	Recyclable	Recyclable / Biodegradable
Microplastic release	Low (*vs.* spun poly)	None
Circularity	High	High

A core innovation in both fabric prototypes, PES and PLA, is the integration of a three-dimensional structure using plain loom, tailored for high-performance use:

- **Breathability & thermal comfort:** The 3D structure improves water vapor permeability within the textile, enhancing moisture management and contributing to thermal regulation during activity;
- **Natural elasticity without elastane:** Both fabrics achieve stretch and mobility through structural design rather than blended elastane fibres, facilitating end-of-life recycling and reducing environmental impact.

To achieve this, an in-depth yarn study was conducted to optimize the fabric structure for mechanical performance and comfort. For the fabric developments, a combination of different filaments was used for PES, while spun yarns were also included for PLA, as detailed in Table 6.

Table 6 Yarn combination for fabric development

Yarn	Structure Code	Warp	Weft	Characteristics
Polyester (EA015000067)	TR1	78 dtex / 72 fil, textured PES	56 dtex / 48 fil + 167 dtex / 48 fil	High flexibility, good air/water vapor permeability, effective stretch
	TR2	78 dtex / 72 fil, textured PES	167 dtex / 48 fil	Good natural elasticity and breathability
PLA **(EA015000109)**	TR3	167 dtex /64 fil, textured PLA	167 dtex /64 fil, textured PLA + Ne 40/2 PLA	High breathability, lower stretch

Yarn	Structure Code	Warp	Weft	Characteristics
	TR4	167 dtex /64 fil, textured PLA	167 dtex /64 fil, textured PLA + Ne 40/2 PLA	High breathability, lower stretch

In case of EA015000067, both TR1 and TR2 structures demonstrated promising mechanical performance, achieving natural stretch without elastane and high air/water permeability, ideal for activewear use. Due to its superior performance in terms of elasticity, TR1 was selected for garment prototype.

Concerning EA015000109, both structures demonstrated very high breathability, but lower elasticity compared to the PES development. Both structures also showed some tendency to snagging, and TR4 was selected due to its better performance in this regard.

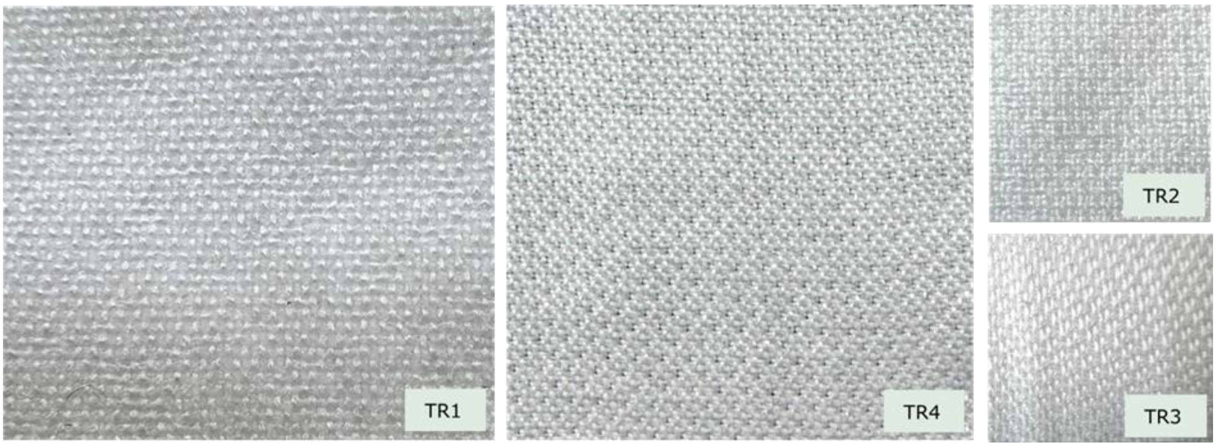

Figure 9 Three-dimensional structure of the fabric prototypes.

2.5.2. Textile spray coating

Both PES (TR1) and PLA (TR4) fabrics were spray coated using the most promising PHA dispersion. As this had to be done on lab scale, and the amount of fabric coated in each batch was limited, only the right front mold for the outfit was coated for each demonstrator. After coating, these were hot-pressed, before the project logo was inkjet printed using the 2D equipment developed within the project, as shown in Figure 10.

Figure 10 Right front molds for the PES (left) and PLA (right) padel outfits, spray coated with the PHA dispersion, and with the project logo inkjet printed.

2.5.3. Demonstrator production

As part of the product development, a functional garment demonstrator was produced to validate both material performance and garment design in a realistic use context. The demonstrator, a skirt, and a top set, targets active use in padel practice, requiring a balance of mobility, breathability, and durability.

3D virtual prototype

The process began with the creation of a 3D virtual prototype (Figure 11), a key tool that enabled fabric testing by evaluating the visual and structural behaviour of the selected 3D textiles (PES and PLA) within the intended garment silhouette. It also supported design and fit validation, including ease of movement, and aesthetic proportions digitally before moving into physical production. Furthermore, the virtual prototype facilitated early optimization by helping to identify and adjust potential design or construction issues, minimizing material waste. Virtual prototyping significantly reduced development time and supported more sustainable sampling by minimizing physical trials in the early stages.

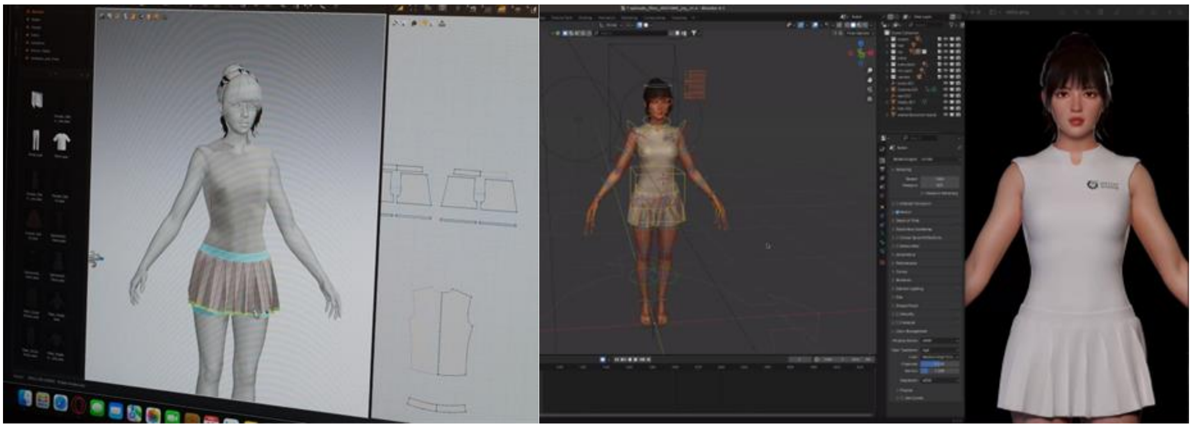

Figure 11 Creation of 3D virtual prototype.

Physical Prototype Development

Following the validation of the digital model, a functional physical prototype of the skirt and top set was produced (Figure 12). This version incorporated the finalized 3D textile structures, as described in previous sections (PES EA015000067 - TR1 and PLA EA015000109 – TR4), along with optimized pattern cutting and assembly techniques. The functionalized fabric spray coated with the PHA dispersion, was applied in the top front, a common area of higher perspiration for women.

The physical prototype successfully confirmed the garment's ability to meet the demands of padel practice, particularly in terms of thermal comfort, stretch, and design.

Figure 12 Physical Prototype: skirt and top set.

3. Conclusions

The work presented in this report successfully demonstrates the feasibility of developing and applying a PHA-based (PHB.E.0) dispersion for textile coating using a spray technique. Through systematic optimization of the dispersion formulation, spray parameters, and post-treatment conditions, a stable and sprayable PHA dispersion was developed and effectively deposited on polyester and cotton fabrics.

Comprehensive characterization using SEM, FTIR, and EDS confirmed the successful deposition and presence of the PHA coating on the textile substrates. Functional tests further revealed that the coated fabrics exhibited hydrophilic behaviour, and that air permeability was reduced, potentially improving protective performance in sportswear applications, while water vapor permeability remained largely unchanged.

For the demonstrator, a circular material strategy was adopted, using recycled polyester filament and PLA, and successfully developed 3D textile structures, a core innovation using plain loom. Although lab-scale coating was successfully achieved, challenges arose during the scale-up process, notably the clogging of spray equipment due to particle aggregation. These issues were attributed to insufficient dispersion stability, highlighting the need for extended mixing and sonication times and improved particle size control in larger batches.

Overall, the results are promising and support the potential of PHA-based coatings for functional textiles, which could replace currently fossil-based coating formulations. However, further optimization and development are required to ensure stable, scalable processing and to fully realize the performance benefits in industrial applications.

The printability of the coated textiles is shown in Deliverable D4.4; their non-toxicity in deliverable D6.1; their biodegradability in D6.2; and their recyclability in D5.2.

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