

Article

Bio-Based Finishes for the Development of Functional Cotton Knitted Fabrics

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Highlights

- Bio-based finishing textiles were developed by valorisation of agro-industrial residues.
- Oregano and thyme extracts produced textiles with antioxidant activity above 85%.
- Rice husk extracts imparted hydrophobicity with WCA values above 140°.
- Excellent UV protection (UPF > 50) was achieved using OS and TS extracts.
- Commercial fixator improved the washing fastness of hydrophobic and UV protection properties.

Abstract

Agro-industrial residues are an important source of bioactive compounds with interesting properties for textile applications. Bio-based formulations were developed by incorporating cationic starch and bacterial cellulose with extracts obtained from agro-industrial residues (oregano (OS) and thyme stems (TSs), spent hops (SHs) and rice husk (RH)), through ultrasound-assisted and water bath extraction. The extract from OS obtained by the water bath showed the highest antioxidant activity (25,272 μ M Trolox), and when incorporated into textiles, it also enabled a high antioxidant activity of 85%. A high antioxidant activity was also recorded in TS extract (21,063 μ M TX), where the functionalized textiles achieved an antioxidant activity of 89%. However, a decrease to 20% in antioxidant activity was recorded after washing. The functionalization with RH extracts enabled the development of hydrophobic textiles, exhibiting static water contact angles above 140, which was maintained after washing. Textiles functionalized with OS and TS extracts achieved excellent UV protection (UPF > 50). Even after washing, the textiles pre-treated with the commercial fixation maintained UPF > 50. Overall, this study demonstrates the potential for valorising agro-industrial residues into functional textiles within a sustainable and circular bioeconomy approach.

Keywords: functional textiles; bio-based formulations; bacterial cellulose; cationic-starch; antioxidants; water repellent; UV-protection



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1. Introduction

The current state of the textile industry raises concerns about environmental sustainability. The low recycling rates, the non-biodegradability of textile materials, the dependence on natural resources, and the use of fossil-based products along the textile value chain are the main concerns [1–3]. In particular, wet-finishing processes such as dyeing and printing account for approximately 72% of the total water consumption in

textile manufacturing. In addition, these stages are associated with the generation of hazardous wastewater, high energy demand, and greenhouse gas emissions [4,5]. To mitigate these environmental impacts, the textile industry has been progressively adopting more sustainable practices, processes, and products [6,7]. Nevertheless, the development and application of certain textile finishes face technical and sustainability challenges.

Natural fibres like cotton, linen or viscose are hydrophilic and tend to adsorb moisture, which makes them comfortable to wear in dry conditions [8,9]. However, for specific applications, including outdoor clothing, workwear, military, and medical textiles, durable water and oil-repellent properties are essential [10,11]. For decades, hydrophobic, oleophobic, and stain-resistant properties have been achieved using long-chain (C_nF_{2n+1} , $n \geq 7$) perfluoroalkyl substances (PFASs), commonly referred to as “forever chemicals”. Due to their bioaccumulative nature and toxic effects on the environment and human health, the European Commission under REACH regulation and regulatory agencies in Japan, China and some states in the U.S.A. have implemented measures to restrict the use of these substances [12–14]. Alternative solutions include silicones, hydrocarbon waxes and dendrimers, which act by forming a thin hydrophobic layer on the fibre surface by reducing the surface energy [15,16]. Although silicone-based finishes provide excellent water repellency, their oil-repellent performance is limited, and their non-biodegradable nature raises concerns [16,17]. Hydrocarbon waxes, renewable (plant and animal waxes) or non-renewable (mineral waxes), have limited durability and stability and low washing fastness [18,19]. Dendrimer synthesis remains complex and presents several limitations, including the instability of certain branches, difficulties in achieving high yields and monodispersity for high-generation dendrimers, and challenges in producing multifunctional dendrimers due to the random distribution of functionalities [20].

The incorporation of antioxidants into textile materials has been associated with well-being and cosmetotextile applications, as these compounds can reduce free radicals, mitigate oxidative stress on the skin, and promote anti-ageing effects [21,22]. Recent studies have explored natural-origin products to impart these functionalities, including propolis and honey [23], eucalyptus essential oils [24] and extracts from the leaves [25], pomegranate peel [26], keratin extracted from chicken feather waste [27] or *Nigella sativa* oil [28]. Besides the antioxidant activity, these bio-based additives can also provide antimicrobial properties, protection against ultraviolet (UV) radiation and colouration to the textile materials [29]. Growing concerns over prolonged exposure to solar radiation and its harmful effects on human health, including premature skin ageing, pigmentation disorders, and carcinogenesis, have increased the demand for textiles with effective UV protection [30,31]. In textiles, UV protection can be achieved through the application of functional finishes that either absorb, reflect, or scatter UV radiation [31,32]. Conventional approaches often rely on inorganic UV absorbers, such as titanium dioxide (TiO_2) and zinc oxide (ZnO), or inorganic UV stabilisers; however, these raise environmental and health concerns [30,31]. In this context, bio-based alternatives have emerged as promising solutions, particularly those rich in natural UV-absorbing compounds such as phenolics, flavonoids, and tannins [26,33]. However, they often exhibit limited photostability, leading to progressive degradation and loss of performance upon prolonged light exposure [34].

Several functional properties can be derived from agro-industrial residues. Those include mainly plant-based materials, such as straws, stems, stalks, husks, peels, and seeds [29,35]. The valorisation of agro-industrial residues through the recovery of bioactive functional compounds and their application in commercial products, including textiles, can both reduce the negative environmental impacts associated with residue generation and offer sustainable alternatives to the petrochemical-based finishing agents [29,36]. In our study, we explored different agro-industrial residues: oregano stems (OSs), thyme stems

(TSs), rice husk (RH) and spent hops (SHs). Aromatic plants from the *Lamiaceae* family, which include oregano and thyme, are recognised by their antiparasitic, anti-inflammatory, antibacterial, and antioxidant activity [37]. The processing of these plants for leaf collection and essential oils extraction generates residues such as branches, stalks, and stems, with low commercial value and that are often discarded [38,39]. The use of oregano and thyme in the textile industry is primarily associated with their essential oils, which are applied to impart antimicrobial and insect-repellent properties [40–42]. Additionally, thyme extracts were used to dye cotton and woollen fabrics [43]. RH is a lignocellulosic biomass obtained during the rice milling process [44]. The annual worldwide rice production is responsible for generating 150 million tons of husk [45]. Due to its composition, which includes silica and high coarseness, RH is unsuitable for animal feed. Furthermore, its resistance to natural degradation and humification, combined with the large quantities generated worldwide, makes this residue a significant environmental pollutant [44,46]. RH has been explored for the production of biochar, activated carbon, and silica-rich fractions, for applications in agriculture, cosmetics, construction materials, composites, and thermal and acoustic insulation [45,47,48]. Specific to textile applications, the development of hydrophobic and flame-retardant fabrics is reported in the literature [49,50]. Common hop (*Humulus lupulus* L.) is one of the ingredients used in beer production, providing its characteristic bitterness, flavour, aroma and microbial stability [51,52]. At the end of the process, significant amounts of SH, consisting of a mixture of beer and hop vegetative matter, are generated at a ratio of approximately 0.2–0.4 kg per 100 L of produced beer [52,53]. SH is mostly regarded as waste and is typically discarded [54]. SH extracts were applied on 95% lyocell and 5% elastane fabric to provide antimicrobial properties against *Staphylococcus aureus* and *Escherichia coli* [55]. From our literature review, no additional reports on the use of SH in the context of the textile industry were identified.

The extracts derived from vegetable and lignocellulosic sources are mainly composed of low-molecular-weight bioactive compounds, including polyphenols, flavonoids, and natural pigments, which can act simultaneously as functional agents and colourants [56]. However, these compounds often exhibit limited affinity with cellulose fibres due to weak intermolecular forces with the hydroxyl groups (–OH) of cellulose and are dependent on absorption/desorption, aggregation, and film-forming mechanisms on the surface of the fibre. Therefore, the natural components are easily desorbed and leached from the fibres surface during washing or friction [56,57]. Strategies to improve the bonding interactions between adsorbed compounds of the natural extracts and the cotton fibre are based on mordenting (pre-, during and post-dyeing/-functionalization), cationization or surface modification strategies [56,58]. In our work, cationic starch (CS) and a commercial fixative were explored as cationizing agents to improve the interaction between the bio-based extracts and the cotton substrate. Bacterial cellulose (BC) was also explored due to its abundance of hydroxyl groups (such as CS) and its reported affinity towards natural dyes, antioxidants and phenolic compounds [59,60]. Although several studies have investigated plant-derived extracts for textile functionalisation, the potential of OS, TS, SH and RH as sources of functional compounds remains unexplored and given the large volumes generated, additional approaches can still be explored. Furthermore, research on fully bio-based finishes is limited, particularly regarding the durability of the imparted functionalities after washing and their validation according to internationally recognised testing standards. Therefore, this work addresses these knowledge gaps by developing and evaluating the performance and durability of bio-based textile finishing formulations derived from agro-industrial residues obtained through sustainable water-based extraction processes by water-bath (wb) and ultrasound-assisted extraction (UAE). Functional textiles

with antioxidant and hydrophobic properties, as well as UV protection, were successfully developed, exhibiting washing fastness for both hydrophobicity and UV protection.

2. Materials and Methods

2.1. Materials

The OS and TS were supplied by Ervital (Mezio, Portugal), a company dedicated to the production and commercialisation of aromatic plants for the preparation of tea infusions and condiments. The RH was provided by Nova Arroz (Oliveira de Azeméis, Portugal), a company that produces, processes, and commercialises rice. The SH were provided by the brewing company Fermentum (Cervejaria Letra, Braga, Portugal). CS was supplied by COPAM (Lisboa, Portugal), a producer and seller of starchy products, using corn as raw material. CEB-UMINHO (Braga, Portugal) provided lyophilised BC. The 100% cotton jersey knitted fabric (150 g/m²) was purchased from TINTEX (Vila Nova de Cerveira, Portugal), already desized and bleached (read-to-dye (RTD)). The commercial fixative Optifix EC liq. C was provided by Archroma manegement llc (Pratteln, Switzerland). All the other reagents were of analytical grade.

2.2. Extraction of Functional Ingredients from Agro-Industrial Residues

OS, TS and RH were ground at a sieve of 1 mm, using a cutting mill (Retsch SM 300 2022, Retsch GmbH, Haan, Germany). The SHs, upon their collection, were preserved by freezing (−15 °C) until further use. Different concentrations (*m/v*) of the agricultural residues were tested: 3%, 5%, and 10%, using water as solvent. In the case of RH, higher concentrations of 15 and 20% were also tested. The wb (OLS26 Aqua Pro 26 L, Grant Instruments, Royston, United Kingdom) extraction was conducted at 50 °C, 100 rpm for 90 min. The UAE (Sonicator Q700-220, QSonica, CT, State, USA) was performed by applying pulses of 40 W, 1 min ON: 1 min OFF for 15 min, in an ice bath. The extracts were filtered using a vacuum system (VP 220, VWR, Leuven, Belgium), and the resulting filtrate was frozen until use. All the extractions performed are detailed in Table 1.

Table 1. Extracts obtained from the different agro-industrial residues by two extraction methods (wb and UAE).

Extracts	Residue	Residue Concentration	Extraction Method
OS_3%_wb	OS	3%	Water bath
OS_3%_UAE	OS	3%	UAE
OS_5%_wb	OS	5%	Water bath
OS_5%_UAE	OS	5%	UAE
OS_10%_wb	OS	10%	Water bath
OS_10%_UAE	OS	10%	UAE
TS_3%_wb	TS	3%	Water bath
TS_3%_UAE	TS	3%	UAE
TS_5%_wb	TS	5%	Water bath
TS_5%_UAE	TS	5%	UAE
TS_10%_wb	TS	10%	Water bath
TS_10%_UAE	TS	10%	UAE
SH_3%_wb	SH	3%	Water bath
SH_3%_UAE	SH	3%	UAE
SH_5%_wb	SH	5%	Water bath
SH_5%_UAE	SH	5%	UAE
SH_10%_wb	SH	10%	Water bath
SH_10%_UAE	SH	10%	UAE
RH_15%_wb	RH	15%	Water bath
RH_15%_UAE	RH	15%	UAE
RH_20%_wb	RH	20%	Water bath
RH_20%_UAE	RH	20%	UAE

2.3. Functional Formulation Preparation

To prepare the CS-based formulations, CS (5% *w/w*) and glycerol (30 wt.% on the dry starch basis) were mixed with the agro-industrial residues extracts (obtained in Section 2.2). The mixture was stirred for 10 min at room temperature. Subsequently, the temperature was increased to 75 °C to promote CS gelatinisation. The mixture was maintained for 45 min at continuous stirring. As a control, water was used instead of the agro-industrial residue extracts to prepare the formulation (CS_Control).

For the BC-based formulations, BC was added at a concentration of 0.5% *w/w* to the agro-industrial residue extracts (obtained in Section 2.2). The dispersion was mixed for 15 min at 450 rpm using an impeller agitator (CAT R100C). After 10 min, glycerol (30 wt.% on the dry bacterial cellulose basis) was added. As a control, water was used instead of the agro-industrial residue extracts to prepare the formulation (BC_Control).

2.4. Application of the Bio-Based Formulations into Textiles

Before functionalization, the cotton knitted fabrics were washed with 2% phosphate standard detergent in a domestic laundry machine (40 °C, 30 min) to remove dirt, dust, and impurities before proceeding with the functionalization tests.

2.4.1. Impregnation with CS Formulation

The knitted cotton fabrics (3.22 ± 0.1 g) were immersed in the formulations at room temperature and submitted to impregnation (Foulard Horizontal/Vertical TYPE HVF 27291, Werner Mathis AG, Oberhasli, Switzerland) at 3 bar and 1 m/min, obtaining a wet-pick up superior to 80%. This process was repeated three times. The textile samples were dried at 100 °C for 5 min, followed by curing at 150 °C for 5 min in a Dryer/Vaporizer (TYPE DHE 51991, Werner Mathis AG, Oberhasli, Switzerland). As a control, the fabrics were impregnated only with the agro-industrial residue extract, without CS (CS_Extract_Control). Three replicates were prepared for each formulation.

2.4.2. Impregnation with BC Formulation

The cotton knitted fabrics were previously cationised (Pre_cat) with Optifix EC liq. C (Archroma managment llc) at 1.25% relative to the fabric weight. The cationisation was performed in a laboratory exhaustion dyeing machine (Labomat BFA, Werner Mathis AG, Oberhasli, Switzerland) at 40 °C during 60 min, with a heating gradient of 1 °C/min. The impregnation process was the same as described in Section 2.4.1. Afterwards, the dried textiles were impregnated with a solution of Optifix EC liq. c. at 15 g/L, repeating the previous impregnation procedure, and thermoset at 150 °C for 5 min. As a control, the fabrics were impregnated only with the agro-industrial residue extract, without BC (BC_Extract_Control). Three replicates were prepared for each formulation. Figure 1 represents the process for the application of BC formulations, BC_Control and BC_Extract_Control into textiles.

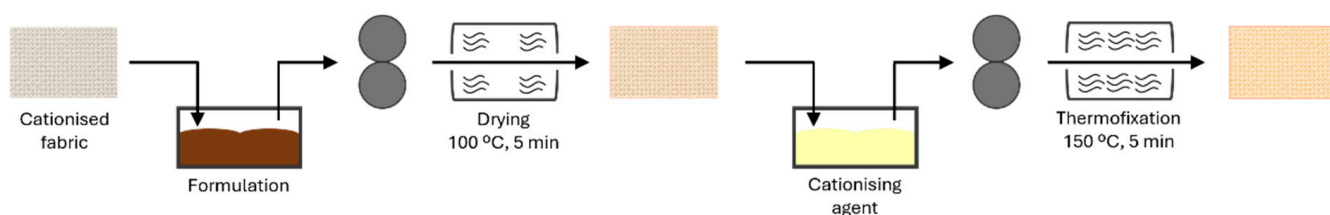


Figure 1. Schematic representation of the functionalization procedure for the application BC formulations, BC_Control and BC_Extract_Control.

In Table 2, the composition of the formulations applied to the textiles is detailed.

Table 2. Detailed composition of the formulations applied to the textile materials.

Formulations	Residue Extract or Water	CS	BC	Textile Material
CS_Control	Water	Yes	—	RTD
BC_Control	Water	—	Yes	Pre_cat
CS_Extract_Control	Agro-industrial residue extract	—	—	RTD
BC_Extract_Control	Agro-industrial residue extract	—	—	Pre_cat
CS formulation	Agro-industrial residue extract	Yes	—	RTD
BC formulation	Agro-industrial residue extract	—	Yes	Pre_cat

2.5. Characterisation Tests

The antioxidant activity of the agro-industrial residues extracts (Table 1) was assessed. The textiles functionalized with the different bio-based formulations (Table 2) were characterised for their functional properties as follows: antioxidant, hydrophobicity, protection against UV radiation, and washing fastness.

2.5.1. UV-VIS

The absorbance spectra of the agro-industrial residue extracts were recorded using a UV-VIS spectrophotometer (UV-2600i ISR-2600Plus, Shimadzu, Kyoto, Japan).

2.5.2. Colour Coordinate Assessment

Colour measurements before and after washing were conducted in a spectrometer (DC Spectro 750, Datacolor, Rotkreuz, Switzerland), using the standard illuminant CIE D65 and an observer angle of 10°. Colour coordinates from CIEL*a*b* colour space, where L* corresponds to lightness, a* corresponds to the transition from green (−a*) to red (+a*), and b* corresponds to the transition from blue (−b*) to yellow (+b*), dE is the colour difference between washed and non-washed textile samples and K/S (absorption coefficient—K—and scattering coefficient—s) referring to colour strength/intensity were accessed.

2.5.3. Phenolic Content of the Extracts

The phenolic compounds quantification was performed by Folin–Ciocalteu method. Initially, a sodium carbonate 4.29% (*m/v*) solution was prepared 12 h before the assay. Then, the Folin–Ciocalteu working solution was prepared in a 1:1 proportion (*v/v*) with distilled water and Folin–Ciocalteu reagent (Panreac Quimica S.L.U., Barcelona, Spain). A calibration curve was performed using gallic acid as a standard. The total phenolic content of the extracts was assessed by adding 111 µL of extract, 111 µL of Folin–Ciocalteu solution (1:1), and 778 µL of sodium carbonate solution 4.29% (*m/v*). The samples were incubated for 1 h at room temperature in the absence of light. Then, the sample absorbance was measured at 760 nm using the UV-VIS spectrophotometer. Two replicas of each sample were prepared.

2.5.4. Antioxidant Properties

The antioxidant properties were evaluated by ABTS scavenging assay. Firstly, ABTS (7 mM; Thermo Fisher Scientific, Waltham, MA, USA) and potassium persulfate (2.45 mM; Sigma-Aldrich, St. Louis, MO, USA) solutions were prepared. The ABTS working solution was prepared by diluting the first solution with phosphate buffer (0.1 M, pH 7.4) until reaching an absorbance of 1.400 ± 0.025 , at 734 nm. The absorbance of the solution was measured by UV-VIS spectrophotometer. A calibration curve was developed using Trolox (TX) as a standard.

The antioxidant potential of the agricultural residue extracts (obtained in Section 2.2) was evaluated by adding 30 µL of the extract to 2970 µL of the ABTS working solution,

followed by incubation at room temperature for 6 min in the absence of light. The results were expressed as TX Equivalent Antioxidant Capacity (TEAC). Three replicates were measured for each different extract.

For the textile samples, 10 mg of each functionalized fabric was immersed in a 5 mL ABTS working solution and incubated for 30 min. Three replicates were prepared for each analysis. Subsequently, the absorbance was measured. The antioxidant activity was calculated using the following equation (Equation (1)), where A_{control} corresponds to the CS_Control/BC_Control absorbance, and A_{sample} to the absorbance of the fabric impregnated with the formulations (CS/BC_Extract_Control and CS/BC formulation).

$$\text{Antioxidant activity (\%)} = \frac{A_{\text{control}} - A_{\text{sample}}}{A_{\text{control}}} \times 100 \quad (1)$$

2.5.5. Hydrophobicity

As a preliminary test, the water repellence of all functionalized textiles was evaluated by measuring the time required for a 30 μL deionised water drop to be fully absorbed. For the textile samples that demonstrated absorption times longer than 1 min, the static water contact angle (WCA) was measured using an optical tensiometer (Theta Flex Auto 1, Biolin Scientific, Gothenburg, Sweden) via the Sessile Drop method. This analysis was repeated 3 times in each sample.

2.5.6. UV Protection

The UV protection factor (UPF) of the functionalized textile samples was determined according to the Australian/New Zealand Standard AS/NZS 4399. In addition to the three textile replicates of each tested formulation, 6 transmittance readings (290 nm and 400 nm) were performed in each textile sample.

2.5.7. Washing Fastness Properties

The washing fastness tested was based on EN ISO 105-B06. The functional fabrics were washed with 4 g/L standard non-phosphate detergent at 40 °C for 30 min, in a laboratory exhaustion dyeing machine (Labomat BFA, Werner Mathis AG, Oberhasli, Switzerland).

2.6. Statistical Analysis

For statistical analysis, Microsoft Excel 365, and GraphPad Prism (version 10.4.1) were utilised. Two-way ANOVA and Sidak's multiple comparisons test ($\alpha = 0.05$) were used to calculate the statistically significant differences in the results between groups. A confidence level of 95% ($p < 0.05$) was considered for the experimental results. Three fabrics were impregnated in the CS and BC formulations, and three replicates were analysed to measure the antioxidant properties, UV protection, hydrophobicity and colour coordinates.

3. Results and Discussion

3.1. Characterisation of the Agro-Industrial Residues Extracts

The antioxidant activity of extracts obtained from the agro-industrial residues using two extraction methodologies, UAE and wb, was evaluated by ABTS method. The results in Figure 2 reveal significant differences in antioxidant activity depending on the type of residue, concentration, and extraction method.

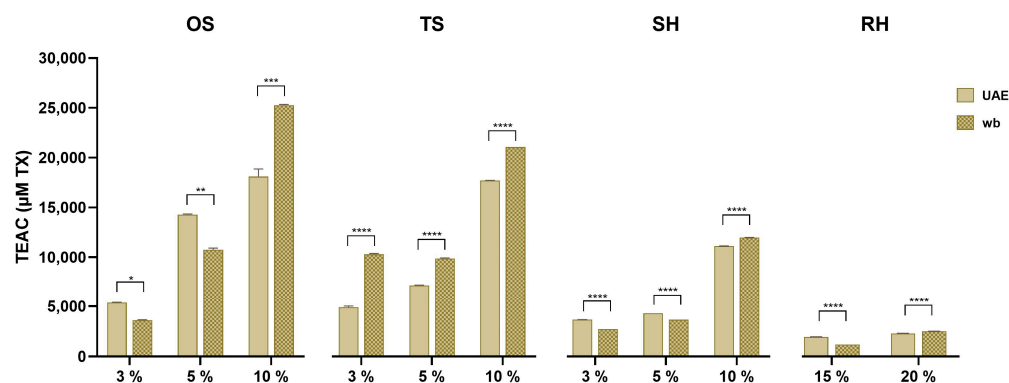


Figure 2. Antioxidant activity of agro-industrial residues extracts obtained from OS, TS, SH and RH by two extraction methods (UAE and wb). Three replicates were analysed, and the results are expressed in TEAC ($\mu\text{M TX}$). The statistical analysis was performed with a two-way ANOVA and Sidak's multiple comparisons test ($p < 0.05$). Statistical significance is indicated as follows: $p \leq 0.05$ (*), $p \leq 0.01$ (**), $p \leq 0.001$ (***) and $p \leq 0.0001$ (****).

Antioxidant activity of the extracts increased with residue concentration, although the extraction efficiency was dependent on both the agro-industrial residue and the extraction method applied. The highest antioxidant activity was reached by OS following wb extraction at the highest residue concentration tested ($25,272 \pm 58 \mu\text{M TX}$), with significant differences compared to UAE ($p \leq 0.001$). When comparing the extraction methods, for TS, SH and RH, the differences are more significant than those recorded for OS. In the case of TS, the wb extraction yielded significantly higher antioxidant activity ($p \leq 0.0001$) than UAE for all the concentrations tested. At 10%, a value of $21,063 \pm 17 \mu\text{M TX}$ was recorded. In the case of SH, wb was significantly ($p \leq 0.0001$) more efficient than UAE at 10%. Due to their minimal antioxidant activity, below $1126 \pm 17 \mu\text{M TX}$ at 3%, 5%, and 10% (Figure S1 in Supplementary Materials), RH extracts were also prepared at higher concentrations of 15% and 20%. However, low antioxidant activity was still observed (below $2512 \pm 5 \mu\text{M TX}$). The UV–VIS absorbance spectra also show differences between the extracts (Figure 3).

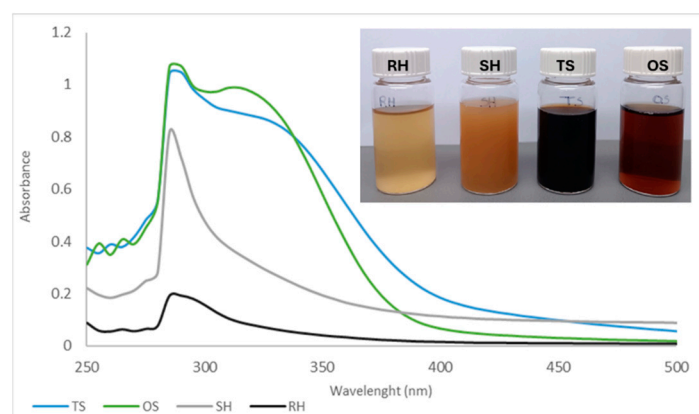


Figure 3. UV–VIS absorption spectra of extracts obtained from TS, OS, SH and RH by wb extraction.

The high antioxidant activity exhibited by the TS and OS extracts is consistent with their UV–Vis spectra (Figure 3), which showed an intense absorption band centred around 290 nm. In addition, the OS extract displayed a strong absorption band at approximately 215 nm, while the TS extract exhibited a less intense band around 320 nm. This spectral feature is commonly associated with phenolic compounds, namely flavonoids, whose aromatic structures and hydroxyl groups are responsible for their strong radical-scavenging capacity [61]. In contrast, this absorption band was considerably less in-

tense in SH and even lower in RH extracts (Figure 3), consistent with their reduced antioxidant activity. These results are also confirmed by the superior total phenolic content (Figure S2 in Supplementary Materials), with OS and TS achieving the highest values (3386.13 ± 94.28 and 3312.80 ± 5.66 mg/L, respectively), contrasting with those obtained from SH and RH (1399.07 ± 27.34 and 537.52 ± 27.15 mg/L, respectively). However, it is relevant to mention that OS and TS extracts have a more intense colouration that also may contribute to the higher absorbance values. Oregano and thyme are aromatic plants belonging to *Lamiaceae* family, known for their phenolic content and antioxidant properties [62].

Mateus et al., (2024) [62] analysed methanolic extracts obtained from Portuguese oregano and thyme. A similar antioxidant capacity (~ 0.58 mmol Trolox equivalents/g residue) was achieved in both plants [62], as in the present work. The antioxidant activity of SH was lower than that observed for OS and TS. Although hops (*Humulus lupulus*) are naturally rich in hop acids, essential oils, flavonoids, tannins, and polyphenols with well-established antioxidant properties, a substantial fraction of these bioactive compounds may have already been extracted, oxidised, or thermally degraded during the brewing process [63]. Furthermore, the chemical composition of SH is strongly influenced by brewing process parameters, including pH control, ion and polyphenol concentrations, homogenization conditions, and oxidative reactions occurring during boiling [54]. RH exhibited the lowest antioxidant activity among all residues, likely due to its highly lignocellulosic and ash-rich composition [38] and considerably lower phenolic content (Figure S2 in Supplementary Materials).

3.2. Antioxidant Properties of the Textiles

The extracts exhibiting the highest antioxidant activity, namely those obtained by wb extraction at the highest tested concentrations (OS_10%_wb, TS_10%_wb, SH_10%_wb, and RH_20%_wb), were selected for the preparation of the bio-based formulations and their subsequent application to textile materials.

The antioxidant activity (before and after washing) of the textiles functionalized with CS_Extract_Control and CS formulations is presented in Figure 4.

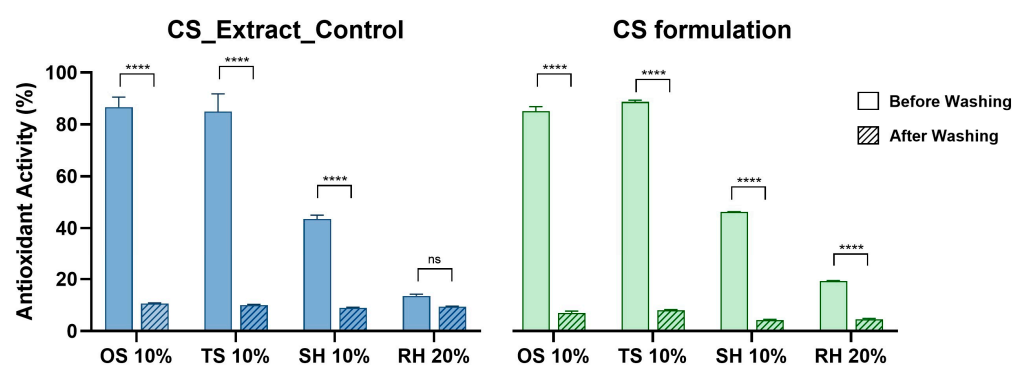


Figure 4. Antioxidant activity (%) of the textiles functionalized with the CS_Extract_Control and CS formulation, before and after washing. Three replicates of the functionalized textiles and respective controls were analysed. The statistical analysis was performed with a two-way ANOVA and Sidak's multiple comparisons test ($p < 0.05$). Statistical significance is indicated as follows: $p \leq 0.0001$ (****).

Before washing, as expected considering the antioxidant activity of the extracts, textiles treated with OS and TS extract formulations exhibited the highest antioxidant activity (85%–90%), with RH showing the lowest among all residues (15%–20%). Since similar antioxidant activities were obtained for CS_Extract_Control and CS formulation, the results suggest that the extracts alone were already effective in imparting antioxidant functionality

to the textile substrate. After washing, a statistically significant decrease ($p \leq 0.0001$) in antioxidant activity was observed for all samples (except CS_Extract_Control of RH), confirming the limited washing durability, even with the addition of CS. This reduction was especially evident for OS and TS, whose activity dropped below 20%, indicating that a large fraction of the antioxidant compounds was not strongly bound to the textile substrate. CS_Extract_Control of RH showed no statistically significant difference ($p > 0.05$) before and after washing, likely due to their already low initial antioxidant activity. CS is reported as a bio-based binder due to its ability to establish electrostatic interactions between the cationic groups and the negatively charged cellulosic fibres [64,65]. In the present study, CS was not effective in promoting a strong fixation of the antioxidant compounds onto the cotton substrate. Wurm et al., 2014 the fixation efficiency of reactive dyes on cotton was dependent on the CS degree of substitution [66] and a direct comparison with our work is not possible, since the Ds of the CS used in this study was not disclosed by the provider for confidentiality purposes. The exploitation of CS to improve the durability of the functional properties or the fixation of natural dyes into cotton substrate is not reported in the literature. The limited effectiveness of CS to improve washing fastness of extract-derived compounds is further supported by the colour measurements (Tables S1 and S2 in Supplementary Materials), since, despite the lower dE (difference before and after washing) observed in CS formulation, the specific values remain higher (superior to 1). For that, CS may be predominantly physically adsorbed rather than covalently bonded to cellulose fibres. Indeed, the release of CS from cotton fibres to a water bath is mentioned by Wurm et al., 2024 [66].

The antioxidant activity (before and after washing) of the textiles functionalized with BC_Extract_Control and BC formulation is presented Figure 5.

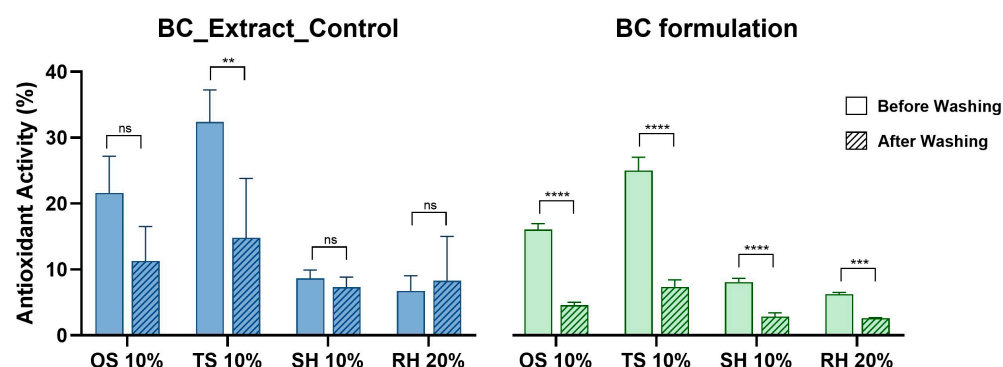


Figure 5. Antioxidant activity (%) of the textiles functionalized with BC_Extract_Control and BC formulation. Three replicates of the functionalized textiles and respective controls were analysed. The statistical analysis was performed with a two-Way ANOVA and Sidak's multiple comparisons test ($p < 0.05$). Statistical significance is indicated as follows: $p \leq 0.01$ (**), $p \leq 0.001$ (***) and $p \leq 0.0001$ (****).

The incorporation of BC into the formulations did not enhance the antioxidant activity of the textiles, nor did it improve the washing fastness of this functionality. Statistically significant reduction in antioxidant activity was observed after washing for textiles functionalized with BC formulations ($p \leq 0.0001$ for OS, TS and SH; $p \leq 0.001$ for RH). When comparing the antioxidant activity, before washing, between the CS_Extract_Control and BC_Extract_Control samples (Figures 2 and 3), the textiles treated with BC_Extract_Control exhibited lower antioxidant activity. These results indicate that the protocol applied for the functionalization with BC_Extract_Control (Figure 1) was not effective in promoting antioxidant functionality onto the textile substrate. Possibly, the immersion of the textile previously impregnated with the agro-industrial residues extracts in the cationiser agent

solution (Figure 1) promoted the release of antioxidant compounds poorly absorbed in the cellulosic fibres. Weak interactions (e.g., hydrogen bonding or physical adsorption) with the hydroxyl groups of cellulose were possibly responsible for the leaching of the bioactive compounds during washing [67]. The phenolic compounds present in oregano and thyme extracts (Figure S2 in Supplementary Materials) may interact with cellulose through hydrogen bonding involving the hydroxyl groups of both the phenolic molecules and the cellulose chains [56]. Similar hydrogen-bonding interactions may also occur with CS (also by an electrostatic attraction) and BC, which are rich in hydroxyl groups and can act as bio-based matrices for the retention of phenolic compounds. For example, thymol and carvacrol, known compounds present in oregano and thymus, contain only one phenolic hydroxyl group per molecule, allowing the establishment of a limited number of intermolecular interactions [68,69]. Since those are weak bonds, justify the significant decrease in antioxidant activity after washing, indicating that the interactions formed between the phenolic compounds, the biopolymers and the textile substrate were not sufficiently strong to ensure durable fixation.

3.3. Hydrophobicity Properties

The hydrophobicity of the textiles, which, in the preliminary test, the water drop took at least more than 1 min to be absorbed, was further characterised by the WCA measurement (Table 3). In both the CS_Control and BC_Control, the water drop was immediately absorbed. Surfaces with a WCA greater than 90° are classified as hydrophobic, and when the WCA exceeds 150°, termed superhydrophobic [19,70].

Table 3. WCA of the textiles functionalized with CS_Extract_Control and CS formulations, before and after washing. Three replicates of each textile sample were evaluated. The statistical analysis was performed with a two-way ANOVA and Sidak's multiple comparisons test ($p \leq 0.05$). Statistical significance is indicated as follows: ns (non-significant $p > 0.05$), $p \leq 0.01$ (**) and $p \leq 0.0001$ (****).

Formulation	CS_Extract_Control			CS Formulations		
	Before Washing	After Washing	Statistical Analysis	Before Washing	After Washing	Statistical Analysis
RH_5%_UAE	135.2 ± 4.0°	145.1 ± 1.0°	**	135.7 ± 2.0°	-	****
RH_10%_UAE	140.1 ± 2.0°	139.1 ± 2.0°	ns	138.0 ± 1.0°	-	****
RH_5%_wb	135.7 ± 2.0°	141.4 ± 3.0°	ns	139.1 ± 0.6°	-	****

Before washing, textiles treated with RH_5%_UAE and wb and RH_10%_UAE exhibited high WCA values, ranging from approximately 135° to 140°, indicating a hydrophobic surface behaviour. The incorporation of CS did not alter the initial WCA values. For the CS_Extract_Control, the WCA values remained high after washing, and no statistically significant differences ($p > 0.05$) were observed (except for RH_5%_UAE, $p \leq 0.01$). In contrast, the CS formulations completely lost their hydrophobicity after washing, as evidenced by the absence of measurable WCA values and by the statistical analysis ($p \leq 0.0001$). Given the hydrophilic nature of CS [8], the incorporation of CS may have promoted water absorption and swelling during washing, facilitating the loss of the surface barrier responsible for the initial hydrophobic behaviour.

The same analysis was performed on the textiles functionalized with BC_Extract_Control and BC formulations (Table 4).

Table 4. WCA of the textiles functionalized with BC_Extract_Control and BC formulations. Three replicates of each textile sample were evaluated. The statistical analysis was performed with a two-way ANOVA and Sidak's multiple comparisons test ($p < 0.05$). Statistical significance is indicated as follows: ns (non-significant $p > 0.05$), $p \leq 0.05$ (*).

Formulation	BC_Extract_Control			BC Formulations		
	Before Washing	After Washing	Statistical Analysis	Before Washing	After Washing	Statistical Analysis
RH_3%_UAE	146.6 $\pm$ 1.0°	143.5 $\pm$ 1.0°	ns	130.2 $\pm$ 4.0°	138.1 $\pm$ 1.0°	*
RH_15%_UAE	142.2 $\pm$ 1.0°	146.0 $\pm$ 2.0°	ns	131.7 $\pm$ 1.0°	138.1 $\pm$ 2.0°	ns
RH_20%_UAE	145.5 $\pm$ 1.0°	144.2 $\pm$ 2.0°	ns	126.3 $\pm$ 2.0°	133.9 $\pm$ 2.0°	*
RH_3%_wb	142.9 $\pm$ 3.0°	142.2 $\pm$ 3.0°	ns	137.4 $\pm$ 0.9°	136.6 $\pm$ 2.0°	ns
RH_5%_wb	144.7 $\pm$ 3.0°	144.9 $\pm$ 1.0°	ns	136.8 $\pm$ 0.7°	139.7 $\pm$ 2.0°	ns
RH_10%_wb	141.6 $\pm$ 1.0°	143.1 $\pm$ 1.0°	ns	136.1 $\pm$ 2.0°	140.6 $\pm$ 3.0°	ns
RH_15%_wb	143.6 $\pm$ 4.0°	146.0 $\pm$ 0.1°	ns	137.3 $\pm$ 4.0°	139.4 $\pm$ 2.0°	ns

The BC_Extract_Control textiles are hydrophobic, with WCA values consistently above 141°, and no statistically significant differences ($p > 0.05$) were observed after washing. When BC was added to the RH formulations, the textiles showed an irrelevant decrease in WCA values, ranging from 126.3 to 137.4°, but remained hydrophobic. After washing, most formulations maintained similar WCA values and remained statistically non-significant ($p > 0.05$), except for BC-formulations with RH_3% and 20%_UAE ($p \leq 0.05$). The improved washing fastness observed for BC_Extract_Control and BC formulations, compared to CS_Extract_Control and CS formulation, is attributed to the pre-treatment with the commercial fixative, which, although not effective in imparting antioxidant properties, proved efficient in producing hydrophobic textiles with good washing fastness. Furthermore, the hydrophobic behaviour does not appear to be directly correlated with the increase in RH extract concentration or the extraction method, since similar WCA values were obtained. Silica-rich fractions, obtained from the pyrolysis of RH and silica nanoparticles, were incorporated in coating formulations to impart hydrophobic properties to textiles [49,71,72]. Moreover, RH is known for its intrinsic low wettability and high-water contact angle values [38]. The hydrophobic behaviour may result from hydrophobic extractable components present in the RH. However, in our study, no elemental analysis of the aqueous extracts was performed and, therefore, the transfer of silica-based compounds from rice husk to the extract and subsequently to the textile substrate could not be confirmed.

3.4. UV Protection of the Textiles

The cotton jersey RTD, CS_Control and BC_Control, recorded UPF values inferior to 15. The textiles functionalized with CS_Extract_Control and CS formulations, which scored a UPF value superior to 15, are presented in Table 5.

Table 5. UPF of the textiles functionalized with CS_Extract_Control and CS formulations, before and after washing. Three replicates of the functionalized textiles were evaluated. The statistical analysis was performed with a two-way ANOVA and Sidak's multiple comparisons test ($p < 0.05$). Statistical significance is indicated as follows: ns (non-significant $p > 0.05$), $p \leq 0.05$ (*), $p \leq 0.01$ (**), $p \leq 0.001$ (***) and $p \leq 0.0001$ (****).

Formulation	CS_Extract_Control			CS Formulations		
	Before Washing	After Washing	Statistical Analysis	Before Washing	After Washing	Statistical Analysis
OS_3%_wb	18.60	11.06	ns	33.10	17.24	*
OS_5%_UAE	25.32	15.70	ns	37.50	23.31	ns

Table 5. Cont.

Formulation	CS_Extract_Control			CS Formulations		
	Before Washing	After Washing	Statistical Analysis	Before Washing	After Washing	Statistical Analysis
OS_10%_wb	22.32	14.70	****	59.10	21.22	ns
OS_10%_UAE	63.47	17.74	ns	30.39	24.75	****
TS_3%_UAE	26.72	17.51	ns	32.95	33.88	ns
TS_5%_wb	43.49	16.12	**	37.37	33.42	ns
TS_10%_wb	52.76	18.86	****	48.09	22.54	**
TS_10%_UAE	57.73	18.08	****	47.54	28.14	***

UPF values superior to 50, and therefore classified as having excellent UV protection, were developed by the incorporation of OS_10%_wb and UAE, TS_10%_wb and UAE. In the case of OS extracts, the incorporation of CS in the formulation (except with OS_10%_UAE) increases the UPF values of the functionalized textiles. As previously discussed, the cationisation capability of CS is reported to enhance the fixation of reactive dyes onto cotton fibres [65,66,73]. In the present study, a similar mechanism is likely to have occurred, since the textiles functionalized with CS formulations presented higher colour intensity (higher K/S values—Tables S1 and S2 in Supplementary Materials). CS as a cationising agent may have introduced positive charges on the anionic cotton fibres surface and thereby enhancing the adsorption and retention of the negatively charged or polar chromophoric compounds present in the extracts. The colouration of the textiles may have contributed to UV protection, since chromophoric structures in natural pigments can absorb and attenuate UV radiation, leading to an overall increase in the UPF values [29]. After washing, a significant decrease in UPF was observed. Only two CS-based formulations containing TS extracts (TS_3%_UAE and TS_5%_wb), maintaining a UPF superior to 30 for which no statistically significant differences were observed after washing ($p > 0.05$). These results are aligned with the lack of effectiveness of CS to improve the washing fastness of functional properties, already noticed for antioxidant properties and hydrophobicity.

The textiles functionalized with BC_Extract_Control and BC formulations, which scored a UPF value superior to 15, are presented in Table 6.

Table 6. UPF of the textiles functionalized with BC_Extract_Control and BC formulations, before and after washing. Three replicates of the functionalized textiles were evaluated. The statistical analysis was performed with a two-way ANOVA and Sidak's multiple comparisons test ($p < 0.05$). Statistical significance is indicated as follows: ns (non-significant $p > 0.05$).

Formulation	BC_Extract_Control			BC Formulation		
	Before Washing	After Washing	Statistical Analysis	Before Washing	After Washing	Statistical Analysis
OS_3%_wb	44.55	61.52	ns	38.96	47.07	ns
OS_5%_UAE	67.07	58.50	ns	63.68	43.50	ns
OS_10%_wb	47.48	64.82	ns	54.04	73.85	ns
OS_10%_UAE	63.11	68.41	ns	69.90	66.43	ns
TS_3%_UAE	51.70	80.78	ns	31.15	44.47	ns
TS_5%_wb	53.44	60.84	ns	64.01	45.21	ns
TS_10%_wb	55.23	71.95	ns	55.42	51.53	ns
TS_10%_UAE	72.81	90.73	ns	71.46	57.83	ns

Almost all textile samples presented in Table 6 exhibited UPF values higher than 50, even after washing, and were therefore classified as providing “Excellent” UV protection, except OS_3% and OS_10%_wb and TS_3%_UAE. Even in these three cases, a UPF superior

to 40 was recorded. The effectiveness of the commercial fixative agent is further supported by the fact that no statistically significant differences ($p > 0.05$) were observed in the UPF values of both BC_Extract_Control and BC-based formulations before and after washing. These results indicate that the washing durability of the UV-protective effect was mainly associated with the commercial cationiser rather than with BC incorporation. According to the supplier's technical datasheet, the commercial cationiser used in this study acts as a cationic fixation agent for reactive and direct dyes, providing excellent domestic laundering fastness. Therefore, the commercial cationiser likely promoted a natural dyeing effect on the cotton textiles by enhancing the interaction between the coloured extract compounds and the fibre surface. This effect is supported by more intense colouration (high K/S values) of the BC_Extract_control and BC formulation compared to CS_Extract_Control and CS formulations.

As discussed above, textiles with UPF values higher than 15 were obtained only with formulations based on TS and OS extracts. These results are consistent with the UV-Vis spectra of the OS and TS extracts (Figure 3), which exhibited the highest absorbance values throughout the UVA-UVB region (280–400 nm) [74]. OS and TS extracts are also richer in phenolic compounds, known to absorb UVA and UVB radiation [61,75]. Additionally, the UV transmittance spectra corroborate the UPF measurements obtained for the functionalized textiles (Figure 6).

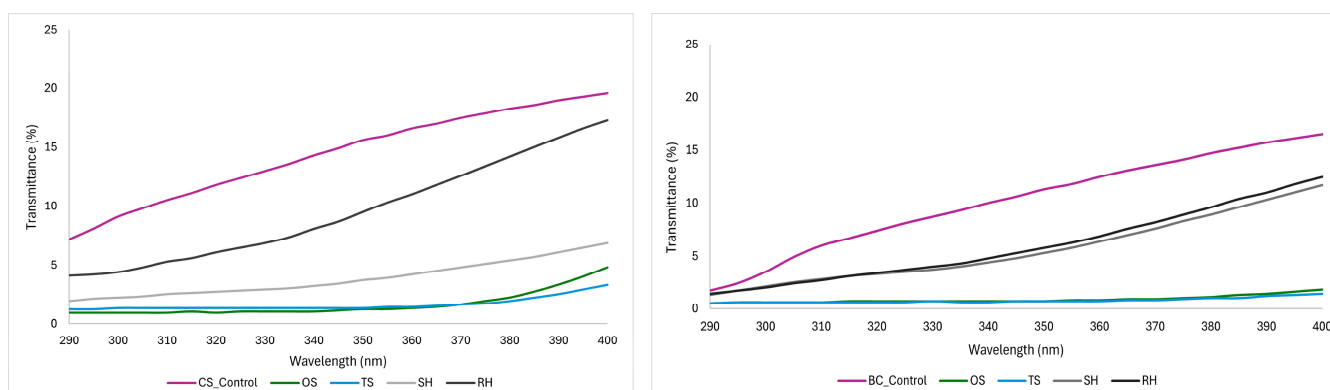


Figure 6. Transmittance spectra of the textiles functionalized with BC formulations (right) and CS formulations (left) and the respective controls.

Textiles treated with OS and TS extracts exhibited the lowest UV transmittance values across the 290–400 nm region, indicating a greater ability to absorb UV radiation [26,74]. In contrast, RH and SH extracts exhibited higher UV transmittance, which is in agreement with their lower UV-shielding performance (UPF inferior to 15). The more intense colouration of the OS and TS extracts themselves (Figure 3) contributed to the development of deeper shades on the functionalized textiles. Consequently, textiles treated with OS and TS extracts exhibited higher colour intensity (K/S values of 1.3514 and 2.7493, respectively) compared to those functionalized with SH and RH extracts (K/S of 0.8500 and 0.2600, respectively).

Textiles with a higher colour saturation and darker shades generally exhibited higher UPF, since they absorb more UV radiation [26].

4. Conclusions

This work demonstrates that different agro-industrial residues can be explored for the development of textiles with distinct functionalities. Among the evaluated residues, OS and TS, obtained by wb extraction, enabled the development of textiles with antioxidant activity superior to 85%. However, these properties were not maintained after washing, regardless of the formulation's composition. Future work should focus on improving

washing durability of antioxidant activity by investigating the use of bio-based and non-toxic crosslinkers, such as citric acid, 1,2,3,4-butanetetracarboxylic acid, chitosan or laccase-mediated approach. Hydrophobic textiles with WCA superior to 140 °C were successfully developed by the incorporation of RH extracts. The incorporation of the commercial fixative agent (Optifix) allowed the maintenance of WCA values above 130° after washing, both in the presence and absence of BC. OS and TS extracts enabled the development of textiles with excellent UV-protective properties (UPF > 50). Due to the fixation effect promoted by the commercial agent Optifix, several textiles maintained excellent UV protection (UPF > 50) even after washing, possibly with great contributions the colouring compounds extracted from OS and TS extracts. Overall, this study highlights the potential of agro-industrial residues as a sustainable source of functional compounds for textile finishing, supporting the transition towards a circular bioeconomy. Nevertheless, further studies, including Life Cycle Assessment (LCA) and biodegradability evaluations in soil and aquatic environments, are required to fully validate the environmental advantages of the developed bio-based textiles over conventional fossil-derived finishing technologies.

Supplementary Materials: The following supporting information can be downloaded at <https://www.mdpi.com/article/10.3390/coatings16070800/s1>, Figure S1. Antioxidant activity, expressed as TEAC (μM TX) of the extracts obtained from RH by ultrasound-assisted extraction (UAE) and water-bath (wb); Figure S2. Total phenolic content of extracts obtained from OS, TS and SH at 10% and RH at 20% by UAE and wb; Table S1. Colourimetric characterisation of CS_Extract_Control before and after washing; Table S2. Colourimetric characterisation of CS formulation before and after washing; Table S3. Colourimetric characterisation of BC_Extract_Control, before and after washing; Table S4. Colourimetric characterisation of BC formulations, before and after washing.

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Abbreviations

The following abbreviations are used in this manuscript:

BC	Bacterial Cellulose
CS	Cationic Starch
LCA	Life Cycle Assessment
OS	Oregano Stems
PFAS	Perfluoroalkyl Substances
Pre-cat	Previously Cationized

RDT	Ready-to-Dye
RH	Rice Husk
SHs	Spent Hops
TEACs	Trolox Equivalents Antioxidant Capacity
TSs	Thyme Stems
TX	Trolox
UAE	Ultrasound-Assisted Extraction
UPF	Ultraviolet Protection Factor
UV	Ultraviolet
wb	Water-Bath
WCA	Static Water Contact Angle

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