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Natural Biopolymer-Based Microcapsules as Sustainable Agents for Hydrophobic Textiles [†]

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Abstract

This study presents the development of hydrophobic coatings for textile applications using natural biopolymers. Natural polysaccharides and waxes in the form of microcapsules were incorporated into a polysaccharide matrix to produce a microcapsule-based coating. Several coating formulations were prepared, incorporating varying concentrations of microcapsules and crosslinking agent (including versions without crosslinker) and subsequently applied to cotton and polyester fabrics using the rod-coating process. The coated fabrics were analyzed in order to evaluate the improvement in hydrophobicity and possible changes in physical properties, while the initial washing stability of the coating was analyzed by determining resistance to one domestic washing cycle. The coating increased the water contact angle from a highly hydrophilic to hydrophobic state (above 120°). After washing, the samples largely retained their hydrophobic properties, with some of them still exceeding a water contact angle (WCA) of 120°. The findings indicate that natural biopolymer microcapsule-based coatings, even without crosslinker, can effectively impart stable hydrophobic properties to textiles, thereby offering a safer alternative to conventional coatings containing per- and polyfluoroalkyl substances (PFAS).

Keywords: PFAS-free; hydrophobicity; coatings; biopolymers; microcapsules; textiles



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1. Introduction

Hydrophobicity is one of the essential functional properties of technical textiles and functional garments, enabling effective protection against water. Hydrophobic coatings are widely used across different sectors, from apparel and upholstery to outdoor and technical textiles, where they can improve the functional performance of products, including sports clothing, waterproof shoes, jackets, backpacks, and tents. For many years, per- and polyfluoroalkyl substances (PFAS) were the dominant materials used to impart strong and durable repellence to textiles. Their unique chemical structure provides outstanding resistance to heat, chemicals, and physical stress [1]. While these coatings are highly effective, their persistence in the environment and potential adverse effects on living organisms have raised concerns [2]. As a result, current research and industrial developments are strongly focused toward the development of safer and more sustainable alternatives. The main challenge remains in creating coatings that combine high hydrophobicity and durable and

stable functionality with environmental compatibility, without compromising on textile properties such as flexibility, breathability, softness and skin-contact comfort. In addition, coatings should be economically feasible and applicable using existing manufacturing technologies to enable widespread adoption. Recent advances in surface engineering have demonstrated that hydrophobicity can be achieved through two strategies, by reducing the surface energy or by increasing the surface roughness by introducing micro- or nanostructures (e.g., lotus leaf effect) [3]. These strategies can enhance the repellency of water in textiles, contributing to improved functional performance. In this study, we report on the development of a hydrophobic coating for textiles that integrates both approaches, while at the same time prioritizing sustainability through the use of natural biopolymers. Hydrophobicity was achieved by combining natural materials with a low surface energy and micro-scale structuring within a single coating system. To achieve that, a combination of polysaccharides and waxes, which exhibit low surface tension, were employed as materials to prepare microcapsules, which were then incorporated into the polysaccharide matrix to produce a coating.

From a sustainability perspective, the natural polysaccharides and waxes used in this study belong to a broader class of biomass-derived polymers, which are being increasingly explored as alternatives to conventional synthetic chemicals and materials. Their use aligns with research directions focused on circular economy and circular material systems, which help reduce the reliance on persistent synthetic chemicals and promote more environmentally responsible material design [4–6]. Although the present work uses commercially available, purified biopolymers rather than waste-derived biomass, the demonstrated functionality of such materials supports ongoing research efforts, aiming towards circular material systems and reduced environmental impact. Future studies could explore the use of non-food or waste-derived biomass as starting materials in the same process, extending the sustainability benefits of the proposed approach.

Microcapsules are small spherical particles, typically ranging from 1 to 1000 μm , widely used in various industrial and scientific fields [7,8]. The microencapsulation process involves enclosing solid, liquid, or gaseous substances within tiny, sealed capsules. The method ensures that the active compound is securely stored inside the capsules, shielded from the environment, and protected against degradation. The capsules enable the controlled release of their contents under specific conditions, such as pH, temperature, light, solvent use, or mechanical force [9–11].

In the context of functional textiles, only a limited number of studies have reported the use of coatings containing microcapsules produced from bio-based polymers. The existing published research describes the use of chitosan, arabic gum, gelatine, polylactid acid, or other bio-based materials for producing microcapsules with natural core substances, with the aim to achieve functionalities like antimicrobial, insect repellent, fragrant, and thermoregulating effects [12–16]. Studies reporting specifically water-repellent behavior using biopolymer-based microcapsules remain limited. One example is a study by Masae et al., which describes a hydrophobic and antimicrobial textile prepared using a coating containing chitosan-based microcapsules dissolved in magnesium sulfate solution [17]. During our literature review, that study emerged as the most promising work concerning bio-based components, as the microcapsules were also produced from chitosan; however, the microcapsules themselves were immersed in an inorganic component, while ours are in water medium. The WCA of the coated cotton was 134° . Furthermore, the resistance to washing of the coated material was not analyzed. Other literature sources focused on the use of microcapsules to achieve multifunctional fabric properties, including hydrophobicity; however, the coatings were only partially bio-based or relied on synthetic components [18–21]. When reviewing the latter publications [18–21] none of the coating sys-

tems described can be classified as fully bio-based. The polyurethane–polydimethylsiloxane (PDMS) phase change microcapsules [19] are partially bio-based, as they incorporate bio-derived shell (chitosan) or core (fatty esters) components, respectively, while relying on non-bio-based fixation strategies. In contrast ethyl-cellulose/silica [20], aluminum-doped zinc oxide AZO-embedded polyurea [21], and pH-responsive styrenic microcapsules [18] are entirely non-bio-based due to synthetic polymer shells, inorganic networks, and/or fluorinated additives. Microcapsule walls range from biopolymers (chitosan) to hybrid organic–inorganic (ethyl cellulose–silica) and fully synthetic structures (polyurea, styrenic copolymers, PU–PDMS), while the cores consist mainly of essential oils and fatty phase change materials. Fabric fixation is achieved either without binders via electrostatic [17] or physical adsorption [18] (chitosan styrenic systems) or through synthetic binders and crosslinking matrices such as silicone emulsions [20,21] or commercial polymer binders [19] which dominate the durability of non-bio-based coatings. In all these studies, hydrophobicity with contact angles exceeding 130° [17,19] or even super hydrophobicity with contact angles exceeding 150° [18,20,21] was reached. While some of these studies, which included synthetic components, did not even perform WCA measurements after washing [17,20,21], others reported retained superhydrophobic values after 10 washing cycles [18], with nearly unchanged WCA values after 25 washing cycles [19]. Nevertheless, it must be emphasized that such performances were obtained with the use of partially bio-based coating systems or systems that were not bio-based at all.

This study presents a fully biopolymer-based microcapsule system that imparts hydrophobic behavior to textile substrates, without the use of fluorinated compounds. A hydrophobic textile coating was developed by incorporating polysaccharide- and wax-based microcapsules into a polymer matrix and applied to natural and synthetic textile substrates with a simple rod-coating procedure which enables scalability. The proposed approach integrates both key strategies for achieving hydrophobicity, surface roughness, and reduced surface energy within a single, sustainable coating system. To critically assess the contribution of chemical crosslinking agents in such systems, coating formulations both incorporating and excluding a crosslinker were investigated, enabling a systematic comparison of their functional performance and initial washing stability. Unlike previous studies, which often relied on the addition of synthetic binders to achieve the desired properties [19,20], this study examines the need and influence of crosslinker addition on the final sample properties and compares it with the developed coating that is crosslinker-free. Given that crosslinkers are often linked to higher toxicity, limited recyclability, impaired fabric handling, and more complex processing [22], such a comparison is essential for understanding their necessity from both performance and sustainability perspectives. Overall, this approach provides a sustainable, safer, and environmentally friendlier alternative to conventional PFAS-based hydrophobic coatings for textiles. By addressing both functional performance and sustainability considerations, it addresses urgent environmental challenges and contributes to the advancement of safe and sustainable hydrophobic solutions for the textile industry that promote healthier conditions for both people and the planet.

2. Materials and Methods

2.1. Materials

A plain weaved, bleached, and mercerized 100% cotton (CO) fabric (185 g/m^2 in weight) obtained from Europrint (Ajdovščina, Slovenia) and polyester (PES) fabric (185 g/m^2 in weight) obtained from Svet Metraže (Ljubljana, Slovenia) were used for the study as substrates for the coating application. Biopolymer-based microcapsules, consisting of beeswax (CAS 8012-89-3), chitosan (CAS 9012-76-4), and arabic gum (CAS 9000-01-5), were supplied from Infokem (Krtina, Slovenia). Chitosan (CAS 9012-76-4, medium molec-

ular weight, $M_w = 950 \text{ kg mol}^{-1}$, $M_n = 250 \text{ kg mol}^{-1}$, $\bar{D} = 3.82$, 76% deacetylated) and acetic acid were purchased from Sigma Aldrich (St. Louis, MO, USA). A crosslinking agent Ruco-Coat FX 8041 was supplied by Rudolf GmbH, Germany. Milli-Q pure water was used for all the experiments in the study.

2.2. Methods

2.2.1. Coating Formulations Preparation

Two concentrations of biopolymer-based microcapsules suspension (5% and 10% of solid content, hereafter referred to as MC1 and MC2) were prepared using a combination of natural biopolymers according to a complex coacervation technique. The exact formulation cannot be disclosed due to confidentiality agreements associated with the project and the company, but the functional contributions of the microcapsule components can be outlined. The microcapsules feature a shell based on natural polysaccharides that provides compatibility with the biopolymer matrix and enhances adhesion to textile fibers, surrounding the hydrophobic natural wax-based core of the microcapsule, responsible for lowering surface energy. The capsule dimensions and composition are designed to ensure sufficient surface exposure following coating and drying, thereby generating surface roughness and a low surface energy, and imparting hydrophobic properties to the treated textiles. For the preparation of the biopolymer matrix, chitosan was dissolved in 1 v/v % acetic acid at a concentration of 2 wt. The initial formulations consisted of the microcapsules suspension and biopolymer matrix (chitosan) in a 1:1 ratio. For comparison, formulations that include crosslinking agent in two different concentrations were prepared, with the highest and lowest concentrations suggested by the supplier (hereafter referred to as C1 and C2). Six coating formulations were prepared, with the names of the coatings and the compounds used listed in Table 1.

Table 1. Coating formulations.

No.	MC1	MC2	Biopolymer Matrix	C1	C2	Abbrev.
1	X		X			MC1
2		X	X			MC2
3	X		X	X		MC1C1
4	X		X		X	MC1C2
5		X	X	X		MC2C1
6		X	X		X	MC2M2

2.2.2. Coating Application

The coatings were applied to CO and PES fabrics, with each sample approximately $35 \times 40 \text{ cm}$ in size, using a rod-coating process using a laboratory coating machine RK Multicoater K303 (RK PrintCoat Instruments, Royston, UK). After the coating application, the samples were dried in a laboratory continuous dryer (Mathis, Oberhasli, Switzerland) at $40 \text{ }^\circ\text{C}$ for 10 min and set in a hot press (Electromechanic Rugelj, Naklo, Slovenia).

2.2.3. Analysis

- Water contact angles (WCAs)

The water repellence of the control (uncoated) and coated samples was evaluated by measuring the WCA. For this analysis, a tensiometer Theta T200 (Biolab Scientific, Gothenburg, Sweden) was used. The static contact angle was determined 5 s after the beginning of the measurement. The average value of three measurements on each sample was reported.

- Resistance to washing

Resistance to washing was determined according to the ISO 105-C06 standard [23] method, using Gyrowash 815 (James Heal, Halifax, UK) apparatus. A simulation of one domestic washing cycle was performed by conducting an A1S test. Samples were immersed in 150 mL of 4 g/L ECE phosphate reference detergent B (SDC Enterprises Limited, Holmfirth, UK) solution and washed for 30 min at 40 °C. After the completed washing, the samples were rinsed twice in 100 mL of Milli-Q water heated to 40 °C for 1 min, squeezed, and dried at room temperature.

- Scanning electron microscopy (SEM)

The surface morphology (the uniformity of the deposit, size and morphological characteristics of microcapsules on a coated fabric) of the samples was analyzed using scanning electron microscope JSM-6060 LV (Jeol, Tokyo, Japan). Prior to observation the samples were coated with a thin layer of gold. The coated samples were observed directly after coating. SEM micrographs were taken at a beam voltage of 10 kV, working distance 16 mm, 30 beam spot size, and a magnification of 50×.

- Mass per unit area

The fabric mass per unit area was determined according to the standard SIST EN 12127:1999 [24]. Five measurements of each sample (cut to size of 100 cm²) were taken and the average value was reported.

- Sample thickness

The fabric thickness was measured according to SIST EN ISO 5084:1996 [25]. Five measurements of each sample were taken, and the average value was reported.

3. Results and Discussion

The aim of this study was to impart hydrophobicity to CO and PES textile materials. Rather than substituting PFAS with other fluorinated compounds, we focused on using only natural biopolymers that are renewable, biodegradable, recyclable, and safe for both the environment and human health. The samples were evaluated for hydrophobicity by measuring the static contact angle with water. A material is considered hydrophobic when water droplets do not spread or wet the material's surface. The transition between wetting and hydrophobicity is indicated by the contact angle of a liquid droplet on the substrate. If the contact angle is below 90°, the substrate is hydrophilic; if it exceeds 90°, it is hydrophobic. To summarize, a higher contact angle corresponds to greater hydrophobicity [26]. Figure 1 shows the results of water contact angle measurements of CO and PES samples coated with all six formulations.

While the uncoated CO and PES fabrics are highly hydrophilic, after the coating application, regardless of the coating formulation, all samples became highly hydrophobic, with the WCA exceeding 90° in all cases. The differences in WCA between the samples coated with two microcapsule concentrations are rather small, with the highest WCA achieved on CO using the lower microcapsules concentration ((MC1) 129.4° for CO and 120.1° for PES), while using MC2 a higher value was achieved for PES (112.9° for CO and 122.6° for PES). The results suggest that using a higher concentration of microcapsules in the formulation does not result in a particularly higher WCA, and that a suspension of microcapsules with lower solid contents in the coating is sufficient to achieve high hydrophobicity. This behavior suggests that critical microcapsule surface coverage may be sufficient to achieve the surface roughness and low surface energy required for hydrophobicity. Beyond this threshold, additional microcapsules mainly embed within the coating rather than further structuring the surface, resulting in only minor changes in WCA. Similar saturation effects

have been reported for other particle-based hydrophobic coatings, where increased particle loading does not improve wetting resistance [27]. Adding a crosslinker to the coating formulation slightly increases the WCA values of all samples, except for the MC1 formulation on CO, which shows lower (however still very high) hydrophobic values after the addition of crosslinker. Other samples exhibit a few degrees higher WCA values after the addition of crosslinker; however no clear correlation can be observed between the concentrations of crosslinker and WCA increase.

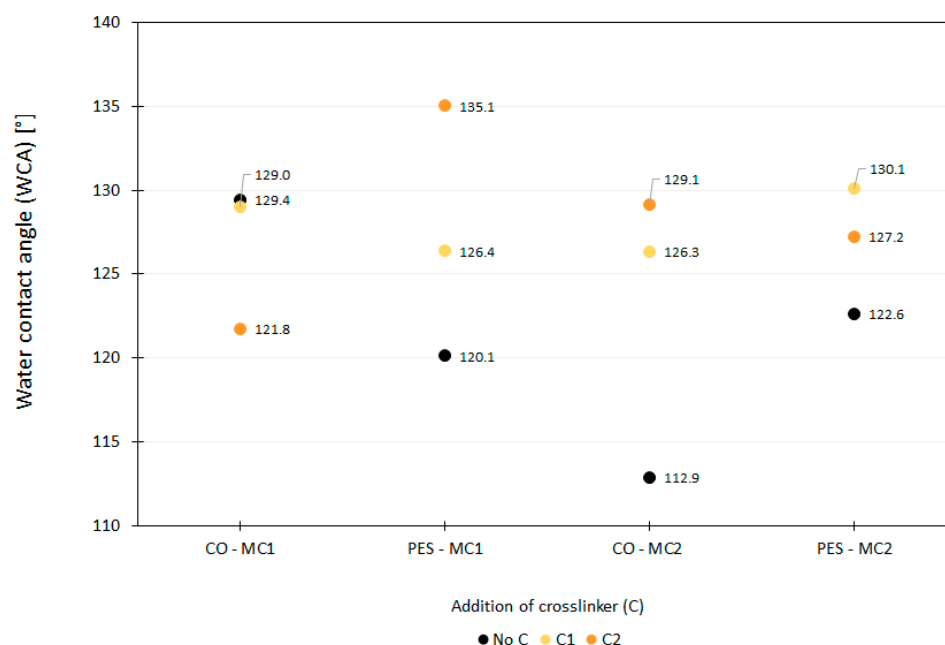


Figure 1. WCA measurements of MC1 and MC2 samples with or without crosslinker at two concentrations (C1 and C2).

As hydrophobicity was achieved even when using a microcapsule suspension of lower concentration, samples MC1, MC1C1, and MC1C2 were further analyzed for resistance to washing to compare if the addition of crosslinker influences the washing stability. The results (Figure 2) showed that even after washing, all samples remained hydrophobic. The addition of crosslinker had no significant effect on washing resistance. Both CO and PES samples without crosslinker (MC1) exceed the WCA value of 120° , which means that the biopolymer-based coating itself shows good fastness to domestic washing. This suggests that coating stability or washing fastness is governed primarily by physical mechanisms rather than the chemical mechanisms provided by crosslinking agents. Mechanical interlocking within the textile substrate structure, combined with interaction between the polysaccharide matrix and fiber surfaces, appears to be sufficient to retain microcapsules during washing. Moreover, the hydrophobic wax-based cores of the microcapsules help maintain water repellence even in the case of possible partial coating loss during washing. These findings underscore the advantage of the developed system, demonstrating that durable hydrophobicity can be achieved without the need for additional chemical crosslinking. The WCA obtained in this work (exceeding 120°), together with stability after standard domestic washing, are comparable with those reported for other PFAS-free and bio-derived hydrophobic textile finishes [17,20,21,28]. Notably, many of those approaches depend on the use of synthetic binders [20], inorganic additives [21], or chemical crosslinking agents [28] to maintain performance. By contrast, the fully bio-based microcapsule system presented here provides comparable and resistant water repellency through a simpler formulation and coating process, highlighting its potential as a sustainable alternative for textile surface

modification. By comparing the hydrophobicity of CO and PES fabrics coated with MC1, we can see that they are both highly hydrophobic, with CO having slightly higher WCA values. This is probably a result of the uniform application of the coating achieved with the rod-coating process. This is further confirmed by SEM micrographs (Figure 3a–d) which show the presence of microcapsules on coated CO and PES samples. The microcapsules are spherical and evenly distributed over the surface of the fibers, indicating a good coverage of the coating. In contrast, the control CO and PES exhibit clean fiber surfaces without any visible particles, confirming that the observed morphological changes are due to the applied coating layer. The deposited microcapsules contribute to the increased hydrophobicity, as supported by the high WCA (Figure 3f). These observations confirm that the applied coating significantly alters the surface morphology and wetting behaviors of the fabrics, leading to a transition from hydrophilic to highly hydrophobic surfaces.

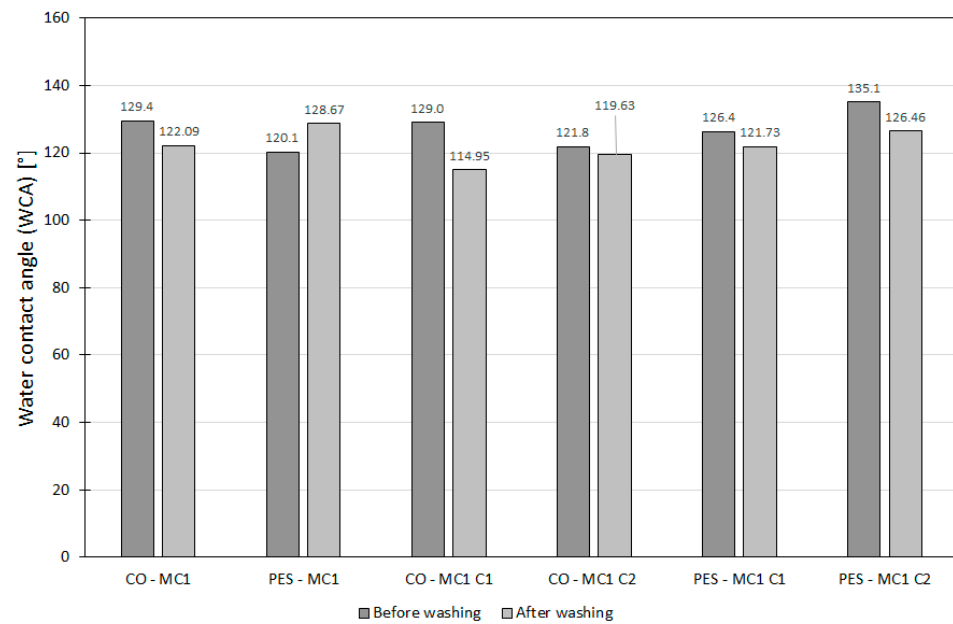


Figure 2. WCA of MC1-, MC1C1-, and MC1C2-coated CO and PES before and after washing.

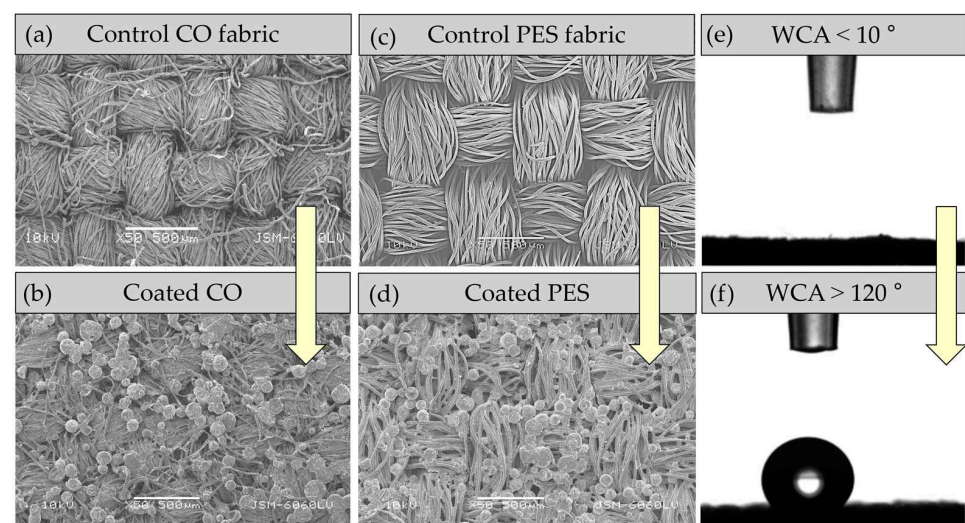


Figure 3. SEM micrographs of (a) control CO fabric, (b) coated CO fabric, (c) control PES fabric, and (d) coated PES fabric at magnification 50 \times and WCA value with the image of droplet of (e) control and (f) coated PES fabric.

For practical applications it is essential that the coating not only improves the textile's functionality but also maintains the fabric's fundamental properties, including softness, hand feel, and overall fabric integrity. Although these characteristics are primarily sensory, changes in mass per unit area and thickness can provide indirect, yet valuable, information about potential alterations in the fabric structure caused by the coating. Therefore, the following section presents the obtained results from mass per unit area and thickness measurements of both untreated (control) and coated samples, enabling the comparison before and after coating application.

As can be seen from the thickness measurements results in Table 2, the coating application resulted in only a negligible increase in the fabric thickness: 0.03 mm for the CO samples and by 0.02 mm for the PES samples. Similarly, as expected, the mass per unit area of the samples also increased following the coating application; however the increase was minimal. In cases of both substrates, coated CO and PES, the mass increased by approximately 30 g/m². These modest changes indicate that the coating did not substantially alter the bulk structure of the fabrics. Although the coated samples were perceived as slightly more textured and stiffer compared with the uncoated ones, due to the presence of the coating, these differences were minimal and did not significantly affect the overall hand feel or flexibility of the samples.

Table 2. Mass per unit area and thickness of control and coated CO and PES samples.

Sample	Mass per Unit Area [g/m ²]	Thickness [mm]
Control CO	180.60	0.35
Coated CO	218.73	0.38
Control PES	152.95	0.41
Coated PES	182.08	0.43

4. Conclusions

This study presents the development of hydrophobic CO and PES fabrics using a fully biopolymer-based microcapsule coating. The coating, uniformly applied by a simple rod-coating process, formed a uniform layer of evenly distributed spherical microcapsules (as observed from SEM images) without causing significant changes in the mass per unit area and thickness of coated samples. The treatment increased the WCA from hydrophilic to highly hydrophobic (>120°), demonstrating hydrophobicity comparable to commercial formulations. Importantly, high hydrophobicity was maintained after washing, even without a chemical crosslinker present, indicating that hydrophobicity can be initially maintained without additional crosslinking chemistry. Unlike conventional fluorine-based coatings, the developed coating is produced from sustainable, safe, and biodegradable biopolymers in the form of microcapsules. While the findings demonstrate that the developed coating represents a viable sustainable alternative to conventional PFAS-based finishes and shows the potential for further optimization towards industrial use, additional studies are needed to assess other sample properties, such as abrasion resistance, long-term stability and durability, and performance under practical use conditions. Overall, this approach provides a promising pathway toward environmentally benign and durable hydrophobic textile finishes derived from renewable sources.

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B.L.; funding acquisition, U.N. and B.L. All authors have read and agreed to the published version of the manuscript.

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