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Structure–property relationships in carnauba wax-based coatings for water-resistant Kraft paper

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Abstract The growing demand for sustainable alternatives to plastic packaging has positioned paper substrates as a key material in the transition toward the use of bio-based solutions for such applications. This study investigates carnauba wax-based coatings emulsified with Tween 80, focusing on how the emulsifier concentration affects the structure, morphology, air permeance, water barrier, and mechanical properties of coated Kraft paper. Scanning electron microscopy (SEM) analysis revealed that the 1:4 (Tween 80:wax, in weight) formulation forms a continuous, defect-free film that fully covers the fibrous paper structure, reducing air permeance from 6.80 to 0.11 $\mu m \cdot Pa^{-1} \cdot s^{-1}$. Higher wax-to-emulsifier ratios (1:6 and 1:7) resulted in severe cracking, incomplete coverage, and an increase in air permeance up to 0.60 $\mu m \cdot Pa^{-1} \cdot s^{-1}$. The 1:4 coating reduced the $Cobb_{60}$ from 104.3 to 21.7 $g \cdot m^{-2}$ and the water-vapor transmission rate (WVTR) from 1151 to 223 $g \cdot m^{-2} \cdot day^{-1}$ at 23 °C and 50% relative humidity. Mechanically, the machine direction tensile strength (~110 N/15 mm) was preserved, while the transverse direction (TD) tensile strength improved from ~ 32.6 to 40.4 N/15 mm. Simultaneously, elongation at break in TD increased from 3.5% to ~ 5%. Overall, the results demonstrate the potential of carnauba wax as a bio-based coating for improving the barrier performance of paper substrates, contributing to the development of more sustainable alternatives to conventional fossil-based paper coatings.

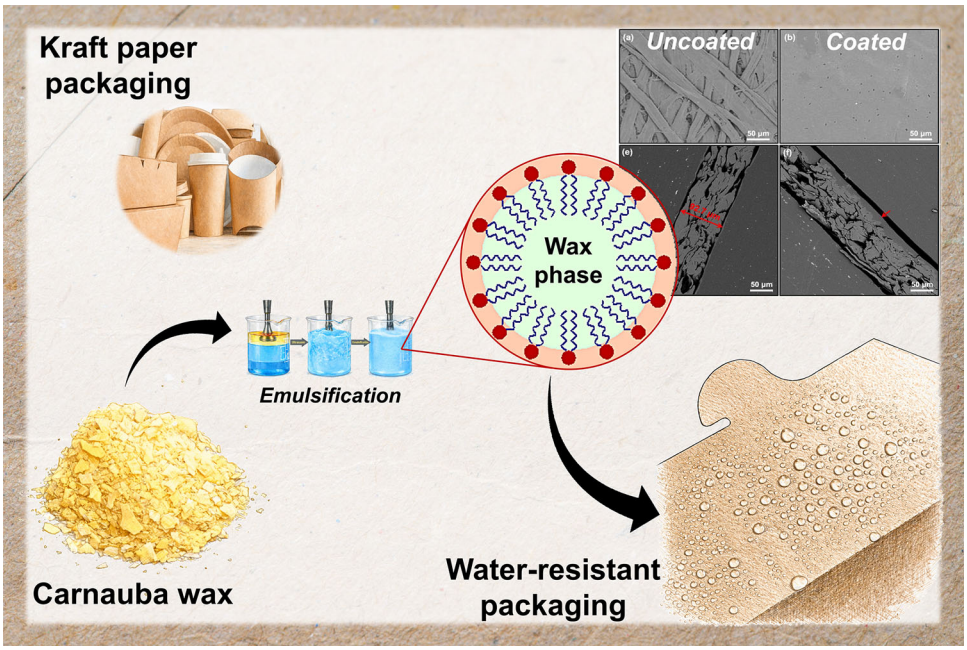
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Graphical abstract



Keywords Water-resistant paper, Bio-based coating, Carnauba wax, Tween 80 emulsifier

Introduction

The combination of different petroleum-based polymers in multilayer plastic packaging yields lightweight and low-cost materials with high oxygen and water-vapor barriers. These characteristics made this type of packaging attractive for sensitive foods, extending the shelf life of the products, avoiding losses throughout the distribution and consumption chains.[1] However, in post-consumption, multilayer plastic packaging is mostly sent to landfills, since there are challenges associated with its recycling, which contravene the principles of the circular economy. Factors such as chemical incompatibility between the used polymers, lack of commercial interest, and the need to collect large volumes of packaging for processing make it difficult to reintroduce these materials into the production system.[2,3]

Cellulosic packaging has the advantages of being produced from renewable resources and having well-established worldwide recycling chains. However, despite being an attractive alternative to fossil-based plastics, the hydrophilic nature and the formation of pores in the structure of these packages make these materials highly susceptible to the permeation of grease, gases, and water vapor.[4,5] The packaging industry has circumvented these characteristics by using fossil-based coatings, which hampers the recycling of paper fibers and does not resolve the economy's dependence on fossil fuels. Therefore, bio-based paper coatings with competitive characteristics compared to conventional plastics are promising solutions as packaging materials with better environmental performance when the coating is compatible with the paper recycling chain.[6]

In this context, wax-based coatings derived from renewable resources have attracted increasing attention because they can provide hydrophobic barrier properties while maintaining compatibility with conventional paper recycling streams. In addition, several studies indicate that natural waxes, such as carnauba wax, are biodegradable and can be integrated into more sustainable packaging systems when applied in thin coating layers on cellulosic substrates. For instance, papers coated with chitosan/carnauba wax emulsions demonstrated improved water and oil resistance and were found to be compostable.[7] Other studies have shown that wax-coated papers can be recyclable and compostable, fitting into a circular economy approach.[8,9]

Among the bio-based materials already studied as paper coatings in the scientific literature, it is worth highlighting polysaccharides (e.g., cellulose, starch, and chitosan), proteins (e.g., soy, pea, and gluten), and lipids (e.g., fatty acids and vegetable waxes).[10–17] The first two groups of molecules have polar functional moieties, which provide low water-vapor barrier properties, leading to the reduction of the shelf life of products sensitive to humidity.[4,5,18] However, the third group is mainly composed of nonpolar molecules, capable of hindering the passage of water vapor through the coated cellulosic packaging.

Matos et al. reported that a water-based emulsion of carnauba wax reduced the water-vapor transmission rate (WVTR) of Kraft paper by 96% at 20 °C/75% RH, effectively sealing the paper's pores.[19] However, dos Santos et al., using a similar formulation under more severe conditions (38 °C/70% RH), found no statistically significant difference in WVTR between coated and uncoated papers, likely due to partial pore coverage and increased water-vapor mobility.[20] They also observed a reduction in tensile strength of approximately 25% in the machine direction and 20% in the transverse direction, attributed to water in the emulsion weakening intermolecular interactions between cellulose fibers. Liu et al. used a Span 80/Tween 80 (1:1) blend to apply microcrystalline wax on a cellulosic substrate, both with and without a prior cationic starch coating.[21] The wax-based coating reduced WVTR by $\sim$ 80%, and by $\sim$ 83% (38 °C/70% RH) when applied over starch. Zou et al. developed coatings based on chitosan and carnauba wax emulsions that, at a 10 $g{\cdot}m^{-2}$ deposition, reduced WVTR by 89% (38 °C/70% RH) and achieved an oxygen transmission rate (OTR) of 94.3 $cm^3{\cdot}m^{-2}{\cdot}day^{-1}$ (23 °C/50% RH).[22]

However, despite these advances, most prior works have focused primarily on performance outcomes, often overlooking a deeper understanding of how formulation variables influence the structural characteristics of the coating layer. In particular, the impact of the wax-to-surfactant ratio on coating formation, integrity, and efficiency has not been thoroughly addressed. Therefore, our study provides a comprehensive evaluation of Tween 80/carnauba wax emulsions applied to Kraft paper substrates. By systematically correlating emulsion composition with coating structure, morphology, physical, water-vapor barrier, and mechanical performance, we aim to bridge this gap and contribute to the development of sustainable, high-performance packaging materials aligned with circular economy principles.

Materials and methods

Chemicals

Polyoxyethylene (20) sorbitan monooleate (Tween® 80) was purchased from Dinâmica Química Contemporânea LTDA (Indaiatuba, Brazil), calcium chloride was purchased from Merck KGaA (Darmstadt, Germany), the type 1 carnauba wax was kindly supplied by Grupo O Boticário (São José dos Pinhais, Brazil), and the Kraft paper was purchased from Spiral do Brasil (São Paulo, Brazil).

Coating of Kraft paper with carnauba wax emulsions

Emulsions were prepared by adding 100 mL of deionized water with Tween 80 and carnauba wax, at weight ratios of 1:4, 1:5, 1:6, and 1:7 (Tween 80:wax). The formulations were designed to achieve a theoretical solid content of 35% (m/v). The mixtures were heated to 90 °C under continuous stirring at 15,000 rpm for 20 min in a high-shear rotor–stator homogenizer (Ultra-turrax T 25, IKA). Subsequently, the emulsions were cooled to room temperature while maintaining a reduced stirring speed of 4,000 rpm. The coatings were applied onto cellulosic substrates using an automatic bar-coating applicator (SVA-B, Mathis Ltd.) with a wet film thickness of 35 μm. The coated papers were then dried in a climate chamber (VC-0060, Vötsch) at 50 °C for 10 min, following procedures adapted from the literature.[19,23,24] The resulting samples were subsequently subjected to characterization.

Characterization of the coated papers

Conditioning

Before the characterization of each paper-based material, all samples were preconditioned below 40 °C and between 10% and 35% of relative humidity for 24 h, followed by their conditioning at 23 °C ± 1 °C and (50 ± 2)% of relative humidity for 48 h, based on the ISO 187 standard.[25] The specimens were prepared and tested under these same atmospheric conditions.

Thickness

The thickness of the coated substrates was determined based on the ISO 534 standard with a micrometer with a resolution of 0.5 μm (model 121, L&Wettre).[26] Five blank test specimens (papers without coatings) and five coated substrate test specimens were evaluated.

Grammage

The grammages of the uncoated and the coated papers were determined based on the ISO 536 standard.[27] The test involved weighing an area of material of approximately 25 cm^2 on a balance with a resolution of 0.1 mg (Practum 224 10 BR, Sartorius). Five test specimens from each sample were evaluated, and the property was expressed as $g.m^{-2}$.

Air permeance

Air permeance is one approach to infer the porosity of paper-based materials. In this test, based on the ISO 5636-5 standard using the Gurley method, a 100-mL air

column is compressed by the weight of a vertical cylinder floating in a liquid (PGH-T, Regmed).[28] The test specimen is placed in contact with the compressed air, and the cylinder falls at a constant rate as the air passes through the material under evaluation. The time required for the volume of air to pass through the test specimen is determined, and its air permeance is calculated. The result is expressed as $\mu m \cdot Pa^{-1} \cdot s^{-1}$.

Scanning electron microscopy (SEM)

The surfaces and cross sections of the samples were evaluated by scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX) techniques (Vega LMU, Tescan) with energy-dispersive X-ray (EDS) (Xplore 30, Oxford) and back-scattered electron (BSE) (LE-BSE, Tescan) detectors. The applied voltage was 10 keV, and the working distance was 15 mm. For the analyses, the coated papers were fixed on aluminum stubs with carbon adhesive and coated with carbon on a metallizer (SCD 050, Balzers). The microphotographs were obtained using the BSE detector. The assay was carried out based on Goldstein et al.[29] theory. The 1:7 formulation exhibited severe macroscopic wax agglomeration after drying, which prevented the formation of a uniform coating suitable for SEM analysis; therefore, this sample was not analyzed.

Fourier transform infrared spectroscopy (FTIR)

Infrared spectroscopy analyses were performed on a Fourier transform instrument (Spectrum 100, PerkinElmer). The attenuated total reflectance (ATR) technique was used. Infrared spectra were obtained following ASTM E573-01 and ASTM E1252-98 standards.[30,31]

Differential scanning calorimetry (DSC)

The melting characteristics of the coatings were evaluated on a differential scanning calorimeter (Discovery DSC 250, TA Instruments), based on the ASTM D3418 standard.[32] Around 10 mg of the materials were analyzed in hermetic crucibles. Initially, their thermal histories were destroyed by heating them to 110 °C, followed by a 5-min isotherm. The samples were subsequently cooled to − 50 °C, followed by another 5-min isotherm, and further heated to 110 °C. Heating and cooling were done at 20 $°C \cdot min^{-1}$. The melting enthalpies of the formulations were compared to that of pure carnauba wax using a relative ratio, normalized by the effective amount of wax present in each formulation. This approach enabled an assessment of the impact of Tween 80 on the melting enthalpy, providing insights into how its presence influences the thermal behavior of the wax-based systems.

***Cobb*$_{60}$**

The water absorption capacity was determined based on the ISO 535 standard,[33] by the difference in mass of the samples before and after contact with water for 60 seconds ($Cobb_{60}$). The measurements were performed on an analytical balance with a resolution of 0.1 mg (Practum 224 10 BR, Sartorius), using specimens with an area of 50 cm^2. The results were expressed in $g \cdot m^{-2}$ at 23 °C. Five test specimens were evaluated per sample.

Water-vapor transmission rate (WVTR)

The water-vapor transmission rate was determined by the gravimetric method, according to ASTM E 96E/E 96 M.[34] Samples with test areas of 50 cm^2 were placed on circular aluminum dishes, which contained calcium chloride as the desiccant agent. The coated side of each sample was placed toward the permeant. The dishes were stored at 23 °C and 50% relative humidity in a climate chamber (Vötsch, model VC-0060) and periodically weighed on an analytical balance with a resolution of 0.1 mg (Mettler Toledo, model AT 400). The WVTR of each specimen, expressed in $g \cdot m^{-2} \cdot day^{-1}$, was calculated with equation (1), in which ''Δm'' stands for weight change, ''t'' for time, and ''A'' for specimen area.

$$WVTR = \frac{\Delta m}{t} \times \frac{1}{A} \quad (1)$$

Mechanical properties

The tensile test of the uncoated and the coated papers was performed based on the ISO 1924-2 standard,[35] using a universal testing machine (5966-E2, Instron) operating with a 1 kN load cell at a speed of 20 mm min^{-1}, and grip separations of 180 mm for machine direction (MD) and 50 mm for transverse direction (TD). Ten 15-mm-wide test specimens were tested for each sample. Their tensile strength (TS, N/15 mm) and their elongation at break (EB, %) were estimated from the average curve of the ten replications. Since the peripheral areas of the coated papers were discharged for the characterization tests, the initial distances between the specimen clamps were 180 mm for the machine direction and 50 mm for the transverse direction.

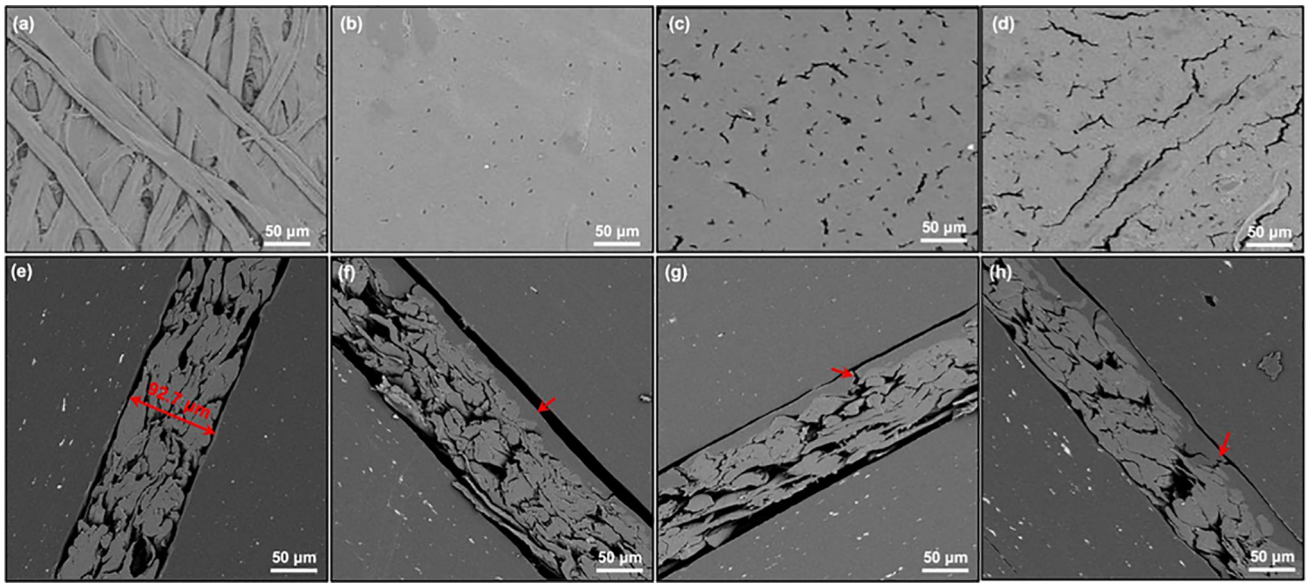


Fig. 1: SEM images (500× magnification) of Kraft paper surfaces and cross sections: (a and e) uncoated paper; (b and f) substrate coated with formulation 1:4; (c and g) substrate coated with formulation 1:5; (d and h) substrate coated with formulation 1:6. The 1:7 formulation was not analyzed due to visible macroscopic wax agglomeration, which prevented proper deposition

Statistics

The collected data were analyzed using ANOVA, followed by Tukey's post hoc test to identify statistically significant differences among the samples at a 95% confidence level.

Results and discussion

Morphology

The SEM images demonstrate the significant impact of emulsifier concentration on the morphology and integrity of the coatings applied to the cellulosic substrates. The uncoated substrate (Fig. 1a) displayed the characteristic fibrous cellulosic paper morphology, with evident air permeance and surface roughness typical of Kraft paper-based materials.[20] Upon application of the 1:4 formulation (Fig. 1b), which contains the highest emulsifier content, a continuous and homogeneous coating layer is observed. The coating effectively masks the fibrous structure, indicating successful film formation with minimal incidence of surface defects, as highlighted in its cross-sectional SEM image (Fig. 1f). Based on the observed coating morphology, this outcome suggests that the emulsifier plays a crucial role in stabilizing the wax dispersion, ensuring adequate wetting and spreading over the substrate.

In contrast, the micrographs for the 1:5 (Figs. 1c and 1g) and 1:6 (Figs. 1d and 1h) formulations reveal a progressive deterioration in coating quality, as indicated in the red arrows. The 1:5 formulation exhibited a noticeable increase in microvoids and fine cracks dispersed across the surface, indicative of partial film rupture likely caused by localized wax aggregation or incomplete coalescence during drying, as inferred from the surface morphology.[36,37] The 1:6 formulation shows even more severe defect formation, with widespread cracking, discontinuities, and incomplete coverage of the substrate. These defects may indicate reduced dispersion stability of the emulsion at lower emulsifier contents, leading to phase separation and inadequate film formation, as observed.

The 1:7 formulation was not shown in the SEM analysis due to severe macroscopic wax agglomeration that was already evident to the naked eye. This observation suggests the existence of a threshold emulsifier concentration required to maintain adequate dispersion of the wax phase: below a critical level, the system loses its ability to maintain a stable dispersion, resulting in poor film integrity both at the microscales and macroscopic scales.[38] Overall, the morphological analysis confirms that sufficient emulsifier content is essential for producing continuous, defect-free coatings on cellulosic substrates. The progressive increase in defects with decreasing emulsifier relative concentration highlights the delicate balance required between the hydrophilic and hydrophobic components in emulsion-based coatings. However, it should be noted that the stability of the emulsions was not directly characterized in this study (e.g., droplet size distribution or colloidal stability measurements). Therefore, interpretations regarding emulsion destabilization are inferred from the observed coating morphology and macroscopic evidence of wax agglomeration.

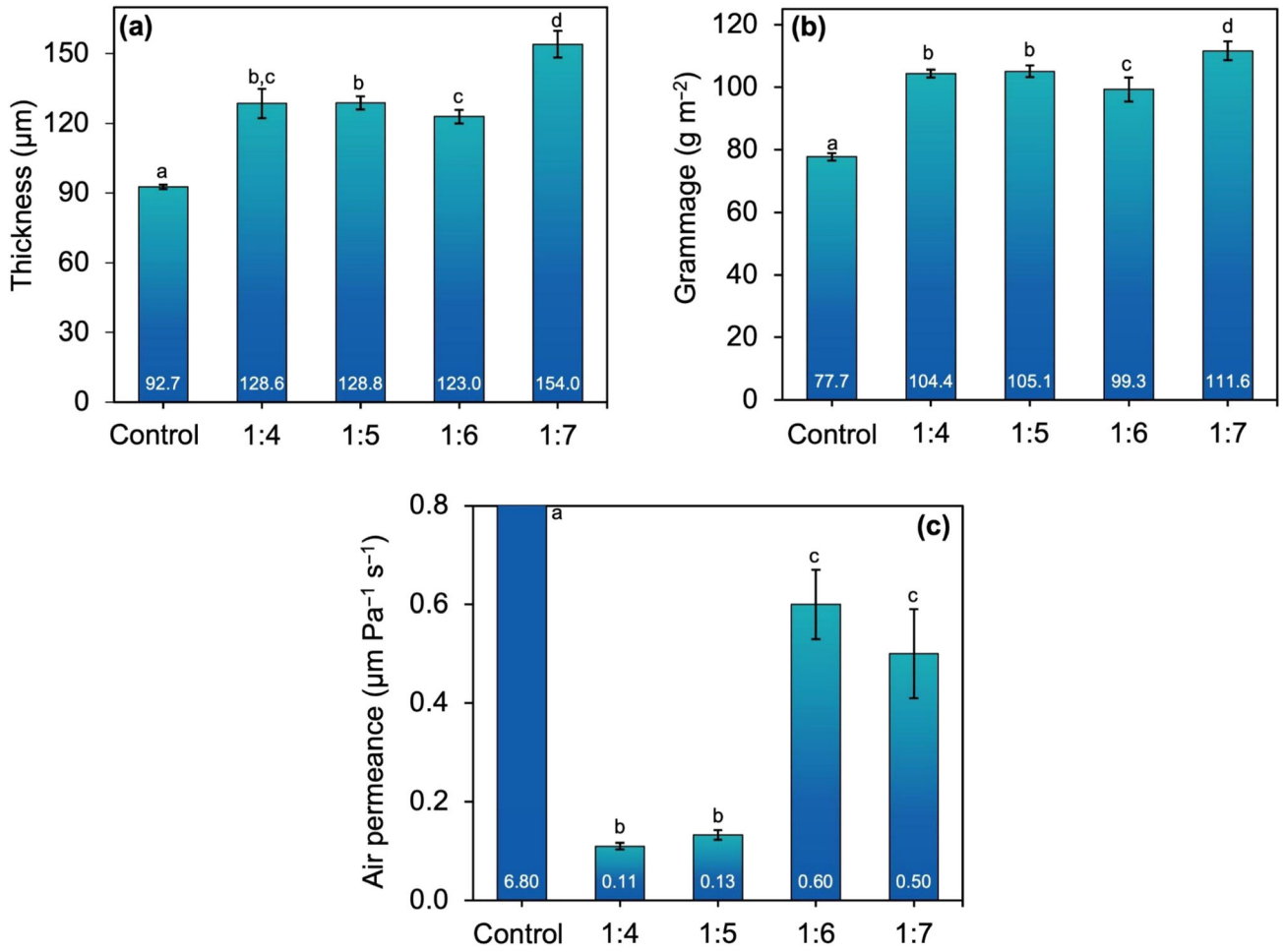


Fig. 2: (a) Thickness, (b) grammage, and (c) air permeance of the control sample (uncoated paper) and the substrate coated with the evaluated wax-based formulations. The values are expressed as mean ± standard deviation (n = 5). Different letters above the bars indicate statistically significant differences between the samples ($p < 0.05$)

Physical properties

Figure 2 demonstrates a clear influence of the emulsifier-to-wax ratio on the physical properties of the coated Kraft paper. A progressive increase in both thickness (Fig. 2a) and grammage (Fig. 2b) is observed as the proportion of wax increases relative to Tween 80, particularly notable in the 1:7 formulation, which exhibited the highest values (154.0-µm thickness and 111.6 g.m^{-2} grammage). This behavior directly correlates with the higher wax loading in the formulation, as lower emulsifier content likely reduces the dispersion efficiency of the wax phase,[39] leading to larger wax domains depositing on the paper surface.

Notably, the formulations 1:4 and 1:5 yielded similar ($p > 0.05$) thicknesses (128.6 and 128.8 µm) and grammages (104.4 and 105.1 g·m^{-2}), indicating that beyond a certain emulsifier concentration, the system reaches a plateau in deposition efficiency. However, the 1:7 formulation shows a statistically significant increase ($p < 0.05$), suggesting that wax agglomeration and defects, already visible macroscopically, lead to the accumulation of thicker and less uniform layers. This observation aligns with the concept of a critical emulsifier concentration reported in emulsion systems,[38,40,41] where the system fails to maintain colloidal stability at low surfactant levels.

In contrast, air permeance (Fig. 2c) shows an inverse trend. The control sample, consisting of uncoated paper, exhibits significantly higher air permeance (6.80 µm·Pa^{-1}·s^{-1}) due to the intrinsic porous nature of cellulosic substrates. Coating with wax-based formulations drastically reduced the air permeance, particularly in the 1:4 and 1:5 samples, which reach minimal values (0.11 and 0.13 µm·Pa^{-1}·s^{-1}). This indicates effective pore filling and surface sealing, consistent with good film formation driven by adequate emulsifier content. However, as the Tween 80-to-wax ratio decreases further (1:6 and 1:7), air permeance partially recovers (0.60 and 0.50 µm·Pa^{-1}·s^{-1}), reflecting the impaired film integrity caused by insufficient emulsifiers.[39] The reappearance of pores and microde-

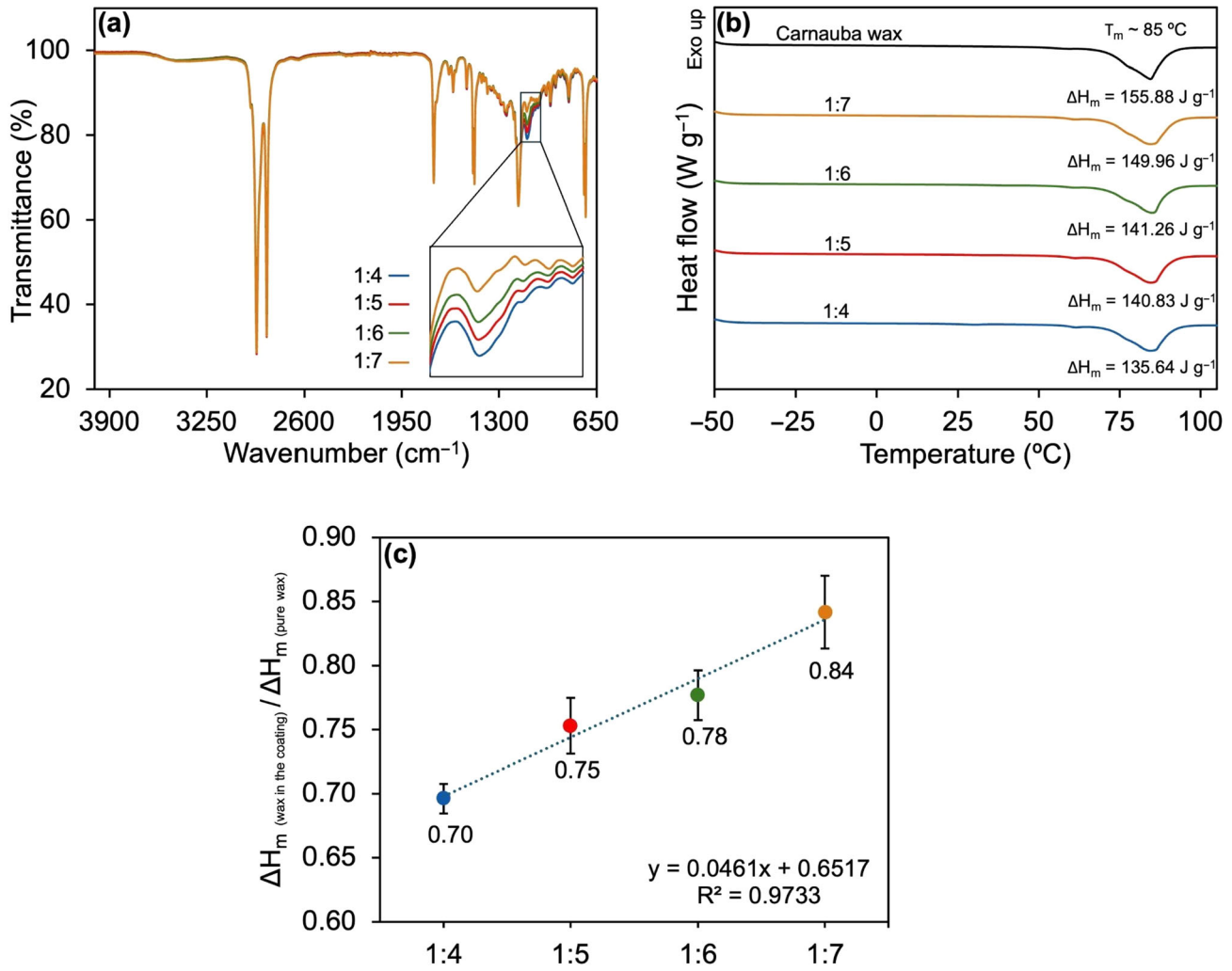


Fig. 3: (a) Infrared spectra of the 1:4 (blue), 1:5 (red), 1:6 (green), and 1:7 (orange) formulations; (b) their thermal behavior and the thermal behavior of the pure carnauba wax; (c) and the ratio of ΔH_m of the wax phase in each coating formulation relative to the ΔH_m of the pure carnauba wax used in the present study, expressed as mean (n = 3)

fects is a direct consequence of wax agglomeration and poor coalescence, as inferred from the SEM observations.

Structure

Among the transmittance bands observed in the FTIR spectra of the coatings after drying (Fig. 3a), several characteristic signals are attributed to the chemical structure of carnauba wax. The prominent bands at 2915 cm^{-1} and 2842 cm^{-1} correspond to the asymmetric and symmetric stretching vibrations of C–H bonds in long-chain aliphatic hydrocarbons, which are the main constituents of wax esters and fatty alcohols.[42–44] Additionally, the band at 1735 cm^{-1} is associated with the stretching vibration of the carbonyl (C=O) group, characteristic of ester linkages present in wax molecules.[45] The signal at 719 cm^{-1} originates from the rocking deformation of methylene (–CH_2–) groups, further confirming the presence of highly ordered alkyl chains characteristic of crystalline wax domains.[46]

It is important to note that the FTIR spectra of the different formulations overlap considerably in the regions associated with the wax structure, indicating that the primary chemical composition of the coatings remains consistent regardless of emulsifier concentration. However, a clear distinction is observed around 1112 cm^{-1}, corresponding to the C–O–C stretching vibration, which is a signature of the ether bonds present in the polyoxyethylene chains of Tween 80 emulsifier.[47] The intensity of this band increases proportionally with the emulsifier content in the formulation, reflecting the incremental incorporation of Tween 80 into the coating matrix. The 1:4 formulation displays the highest intensity at 1112 cm^{-1}, while the 1:6 and 1:7 formulations exhibit significantly lower

intensities, consistent with their lower emulsifier content.

Figure 3b presents the DSC thermograms of the pure carnauba wax and its formulations with increasing proportions of wax (ratios 1:4 to 1:7, Tween 80: wax). As the proportion of carnauba wax increases in the formulation, a clear rise in the melting enthalpy (ΔH_m) is observed, progressing from 135.64 J g^{-1} (1:4) to 155.88 J g^{-1} in the pure wax. This trend reflects the growing crystalline content attributable to carnauba wax in the mixtures. Figure 3c shows the relative ΔH_m of carnauba wax in each formulation, expressed as a fraction of the ΔH_m of the pure wax used in this study. To determine this, the ΔH_m of the wax fraction in each coating was normalized by the ΔH_m of pure carnauba wax, providing an estimate of the crystalline phase preserved in the presence of varying amounts of emulsifier.

Even after normalization, formulations with higher Tween 80 content (e.g., 1:4 and 1:5) exhibit lower relative enthalpies compared to those with higher wax content (e.g., 1:6 and 1:7). This behavior can be attributed to the disruptive effect of Tween 80 on the crystalline structure of carnauba wax. Tween 80 is a nonionic surfactant with amphiphilic character, capable of interfering with the self-assembly and ordered packing of wax molecules during solidification. As a result, the crystallinity and molecular order within the wax phase are reduced, leading to lower energy requirements for melting, i.e., decreased ΔH_m. This interpretation is supported by Zhang et al.,[48] who performed X-ray diffraction analysis on carnauba wax incorporated with 30% tween 80 and found a significant reduction in crystallinity, from 68.3% in pure wax to 58.1% in the modified system. The authors propose that polysorbates, such as Tween 80, cannot fully integrate into the crystalline zones, also being localized in the amorphous regions, acting as impurities or plasticizers. This leads to decreased crystalline content without significant changes in crystallite size. Therefore, even when the wax content is accounted for, the structural disorder introduced by Tween 80 results in a net decrease in ΔH_m, consistent with the thermal data observed in the present study.

These changes in wax crystallinity may also influence the barrier performance of the coatings. Highly ordered crystalline domains typically contribute to lower permeability by creating more tortuous diffusion pathways for water molecules.[6,49] However, excessive disruption of the crystalline structure by surfactants may reduce this effect by increasing the proportion of amorphous regions within the coating.[50–52] In the present study, the DSC results suggest that Tween 80 partially disrupts the crystalline packing of carnauba wax, which may influence how the wax phase organizes within the coating layer and fills the porous structure of the paper substrate. Consequently, the balance between crystalline order and dispersion stability appears to play an important role in determining the overall water resistance and water-vapor barrier performance, as discussed subsequently.

Interactions with water

Figure 4a displays the water uptake capacity ($Cobb_{60}$), while Fig. 4b shows the water-vapor transmission rate (WVTR) of the coated samples and the control (without coating). The results reveal a clear correlation between emulsifier content and interactions with water. The 1:4 formulation exhibited the best performance, with the lowest $Cobb_{60}$ (12.9 g·m^{-2}) and WVTR (154.3 g·m^{-2}·day^{-1}) compared to the control sample ($p < 0.05$), reflecting an effective moisture

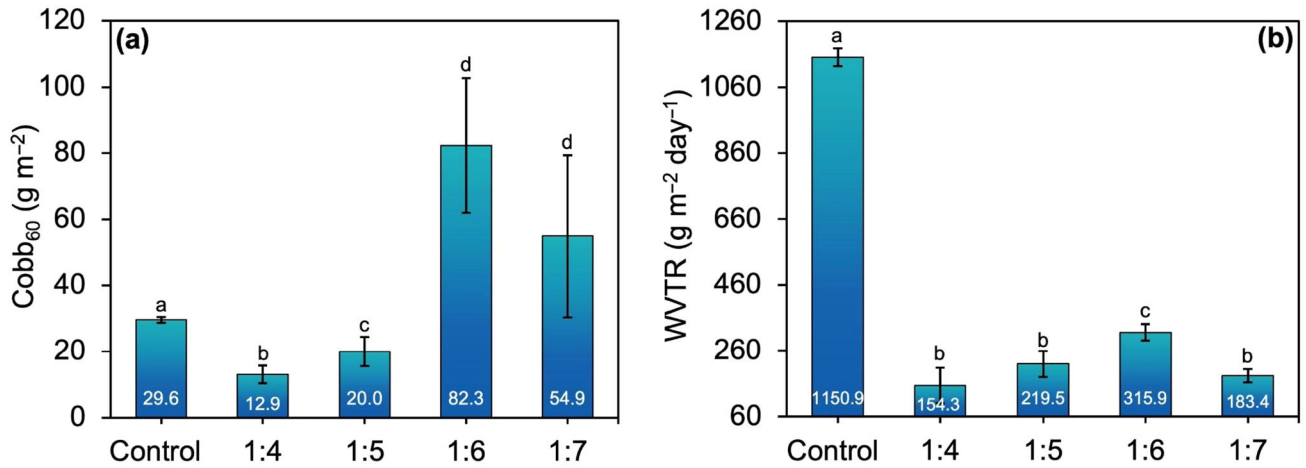


Fig. 4: (a) Water uptake capacity, expressed as the mass of water absorbed per day using the $Cobb_{60}$ method, and (b) water-vapor transmission rate for the control sample (uncoated paper) and the substrate coated with the evaluated wax-based formulations. The values are expressed as mean ± standard deviation ($n = 5$). Different letters above the bars indicate statistically significant differences between the samples ($p < 0.05$)

barrier. This aligns with SEM micrographs, which demonstrate a continuous, defect-free coating that efficiently covers the cellulose fibers, minimizing air permeance. A similar trend was reported by Ning et al.,[40] where increasing surfactant content improved film continuity on wood surfaces, reducing surface roughness.

The 1:5 formulation also provides substantial barrier improvements, albeit with slightly higher $Cobb_{60}$ and WVTR than 1:4, which correlates with the appearance of minor cracks and microvoids in the coating, as evidenced in the SEM images. However, the barrier performance deteriorates considerably in the 1:6 and 1:7 formulations, where $Cobb_{60}$ rises to 82.3 and 54.9 $g{\cdot}m^{-2}$, and WVTR to 315.9 and 183.4 $g{\cdot}m^{-2}{\cdot}day^{-1}$, respectively. These results suggest that both coating integrity and formulation composition influence water transport through the coated paper. Interestingly, the WVTR of the 1:7 formulation was statistically similar to those of the 1:4 and 1:5 coatings ($p > 0.05$) despite its poorer resistance to liquid water. This behavior may be related to the combined effects of coating thickness and structural heterogeneity. The 1:7 formulation produced thicker coatings, which may partially reduce vapor transmission due to the increased diffusion path length.[49,53] However, SEM observations indicate the presence of wax agglomerates and structural defects, which could generate heterogeneous diffusion pathways for water vapor. Therefore, the final WVTR behavior likely results from the competing effects of coating thickness and defect density within the coating layer. It should be noted that this interpretation is based on indirect morphological evidence, since WVTR was not normalized with respect to coating grammage or thickness, and the microstructural continuity of the coatings was not quantitatively evaluated.

In addition to morphological factors, the thermal analysis (Fig. 3) indicates that the presence of Tween 80 partially disrupts the crystalline organization of carnauba wax, as evidenced by the reduction in melting enthalpy (ΔH_m). Since highly ordered crystalline domains typically reduce permeability by limiting molecular mobility and increasing diffusion tortuosity,[54] partial disruption of the wax crystalline structure may also influence the barrier performance of the coatings. Thus, the observed WVTR values likely reflect the combined influence of coating morphology, thickness, and the degree of crystalline organization within the wax phase.

These observations align with studies on bio-based coatings for paper, where insufficient coating integrity resulted in the recovery of air permeance and compromised barrier properties. Du et al. demonstrated that only well-distributed, defect-free regenerated cellulose coatings led to substantial reductions in water-vapor permeability and air permeance,[55] while Dhakane-Lad et al. showed that optimized beeswax–chitosan ratios were critical to achieving low air permeance and water absorption.[56] Similarly, Ning et al. reported that falling below the critical emulsifier concentration leads to wax destabilization, aggregation, and impaired film formation.[40] Overall, these results highlight that barrier efficiency is governed not merely by the amount of coating material but by achieving stable dispersions that form continuous and defect-free layers, a condition met only in the 1:4 and partially in the 1:5 formulations of this study.

Mechanical properties

The tensile properties shown in Fig. 5a reveal a clear anisotropy between the machine direction (MD) and

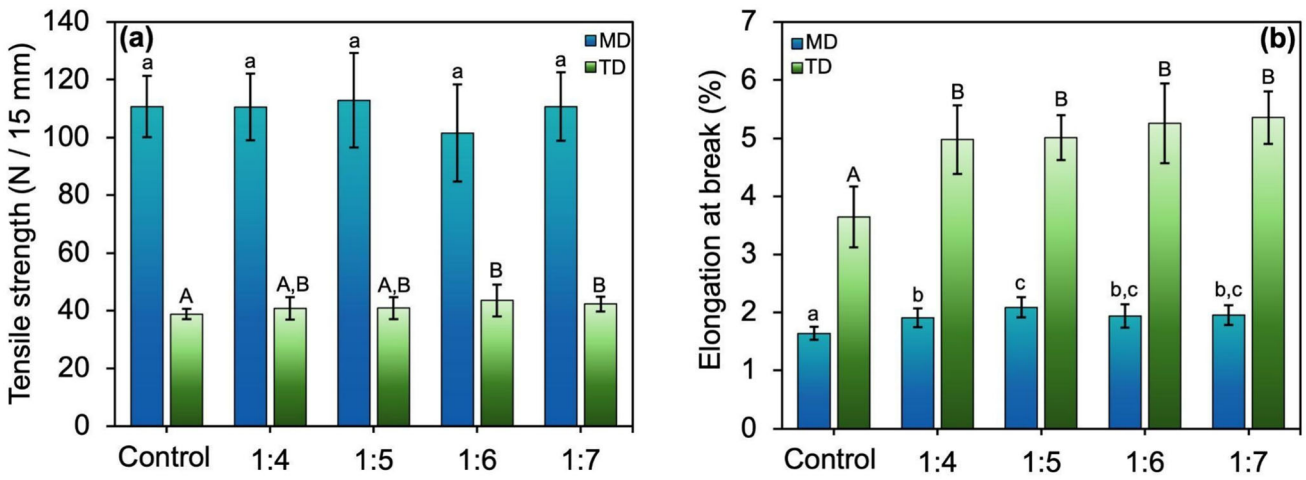


Fig. 5: (a) maximum tensile strength in the machine direction (MD) and transverse direction (TD) as a function of formulation, and (b) elongation at break in the machine direction (MD) and transverse direction (TD) as a function of formulation. The values are expressed as mean ± standard deviation ($n = 10$). Different letters above the bars indicate statistically significant differences between samples ($p < 0.05$)

transverse direction (TD), characteristic of uniaxially processed films. The MD tensile strength (~105–115 N/15 mm) remained stable across formulations, indicating that compositional modifications did not compromise the Kraft paper structural integrity in the dominant load-bearing direction. This behavior is consistent with the structural orientation of cellulose fibers along the machine direction, which primarily governs the mechanical resistance of the paper substrate. Similarly, Wu et al. showed that crosslinking in coated paper significantly enhanced tensile properties without detrimental effects on process-aligned mechanical performance.[57]

In the TD, a modest yet significant increase ($p < 0.05$) in tensile strength for formulations 1:6 and 1:7, compared to control sample, indicates improved stress transfer capacity perpendicular to the orientation. A possible explanation can be partially attributed to better interaction between matrix components or microstructural alignment, comparable to the reinforcement mechanism observed in the crosslinked chitosan-coated Kraft papers reported by Shankar et al.[58] In their system, strong interactions and restricted fiber mobility via crosslinked alkyl chitosan networks were responsible for the improved performance. While the magnitude of absolute tensile values differs due to the distinct nature of paper-based substrates versus flexible films, the underlying mechanism is conceptually aligned, i.e., the reinforcement through interfacial interactions.

Figure 5b illustrates the elongation at break in the MD and TD for the different formulations. The increase in elongation at break in the TD (from ~ 3.5 to ~ 5% for formulations with higher wax content) reflects a desirable shift toward toughness without sacrificing MD strength. This mirrors the design rationale seen in both Wu et al.[57] and Shankar et al.,[58] where balancing strength and stretchability was essential. Notably, Shankar et al. also reported that excessive crosslinking led to increased brittleness,[58] a trend not observed in the present system, suggesting that the formulation avoids over-constraining the polymer network. Therefore, the results indicate that the wax-based coatings do not introduce significant embrittlement to the paper structure while maintaining the intrinsic mechanical integrity of the substrate. Although similar trends have been reported for coated paper systems in the literature,[58] the mechanisms responsible for these changes were not directly investigated in the present study.

Although the emulsions in this study were prepared using high-shear homogenization and applied by laboratory-scale bar coating, the formulation concept may be transferable to conventional paper coating technologies used in industrial production. In industrial environments, wax-based emulsions are commonly applied through processes such as size press, blade coating, or curtain coating,[6,59] which operate under different hydrodynamic and shear conditions. The relatively high solid content of the formulations used here may require adjustments in viscosity and droplet size distribution to ensure stable processing under such conditions. In addition, the homogenization step used in this work represents a laboratory approach to obtain stable dispersions, whereas industrial processes typically rely on rotor–stator mixers or high-pressure homogenizers to achieve similar emulsification.[59] Therefore, while the results obtained here demonstrate the functional potential of carnauba wax emulsions as barrier coatings for paper substrates, further studies focusing on emulsion rheology, long-term stability, and process compatibility would be necessary to fully assess the scalability of the system for commercial coating operations.

Conclusions

Carnauba-wax-based coatings can improve the water barrier properties of paper substrates without jeopardizing their mechanical characteristics. By applying them as emulsions, the study highlighted that the emulsifier plays a crucial role in stabilizing the wax dispersion. SEM analysis evidenced inadequate film formation and increased incidence of failures, such as cracks and pinholes, at lower emulsifier contents. The 1:4 formulation, which had the largest amount of emulsifier, reached 154.3 $g \cdot m^{-2} \cdot day^{-1}$ of WVTR (23 °C/50% RH), 0.11 $\mu m \cdot Pa^{-1} \cdot s^{-1}$ of air permeance, and 12.9 $g \cdot m^{-2}$ of $Cobb_{60}$, which correspond to reductions of 81%, 98%, and 57%, respectively, regarding the uncoated paper. Overall, the results emphasized that barrier efficiency is not determined solely by the amount of deposited coating material but rather by achieving stable dispersions that form continuous and defect-free layers. The progressive increase in defects with decreasing emulsifier content highlights the delicate balance required between the hydrophilic and hydrophobic components in emulsion-based coatings.

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Author contributions G.H.M. conceived the study, performed the experiments, and wrote the first draft of the manuscript. D.S.O. assisted with experimental design and data acquisition. P.F.J.B. contributed to sample preparation and characterization. D.I. performed data analysis and contributed to interpretation of the results. R.P.V. supervised the project, secured funding, guided the scientific strategy, and critically revised the manuscript. All authors discussed the results and reviewed and approved the final version of the manuscript.

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Data availability The data supporting the findings of this study are available from the corresponding author upon reasonable request.

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