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Posted Date: 23 May 2025

doi: 10.20944/preprints202505.1822.v1

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UV-Shielding Biopolymer Coatings Loaded with Bioactive Compounds for Food Packaging Applications

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Abstract: Four natural bioactive compounds with UV-absorbing properties – curcumin, quercetin, caffeic acid, and hymecromone – were incorporated into pectin-based coatings deposited on oriented polypropylene (OPP) to develop packaging films with UV-shielding capabilities. The effects of both bioactive compounds (used individually or in combination) and coating thickness (δ = 0.12 – 1.2 μm) on the optical properties (UV-Vis transmittance and haze) of the coated OPP samples were investigated. Coating deposition enhanced the UV-barrier properties in relation to the type of bioactive compound, following the order: caffeic acid > hymecromone > curcumin > quercetin. Regardless of the type of bioactive compound used, no significant changes were observed in clarity and haze of OPP. Additionally, a greater coating thickness resulted in lower UV-light transmittance of coated PP films. Although the combination of hymecromone and caffeic acid did not exhibit a synergistic effect, it demonstrated an additive benefit, effectively broadening the wavelength range of UV protection in the final packaging materials. While this study highlights that a performance gap remains compared to commercially-available UV-shielding materials, it underscores the potential of replacing synthetic UV-absorbing additives with natural compounds through coating technologies rather than masterbatch incorporation.

Keywords: plastic materials; natural molecules; optical properties; food preservation

1. Introduction

Food exposure to UV radiation can cause light-induced degradation of sensitive compounds, including proteins, lipids, vitamins, and pigments, possibly leading to undesirable chemical alterations, such as off-flavors, discoloration, lipid oxidation, and reduced nutritional content, all of which contribute to a decrease in food shelf life, quality, and safety [1,2].

Packaging can play a pivotal role in the protection of foods from UV radiation. For example, aluminum (as a foil in multilayer configurations or as a vacuum deposited thin layer) acts as a total barrier against UV as well as visible light, while cellulosic materials offer near-complete protection. However, these materials lack the so-called "see-through" capability, that is, they do not allow a clear display of the product through the package. Hence, there is growing interest in the development of transparent materials able to grant efficient protection from potential UV damages [3,4].

Polyolefins, such as polyethylene (PE) and polypropylene (PP), while offering good to excellent transparency, lack inherent protection against UV radiation. For this reason, UV absorbers of synthetic origin are incorporated into the polymer during extrusion, which serves the twofold purpose of protecting both the packaging material and the packaged food from UV light [5,6].

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Currently, different types of UV-absorbers can be used. Phenolic-type UV absorbers are divided into two main groups: those forming an O-H-O bridge, such as salicylates, 2-hydroxy benzophenones, 2,29-dihydroxy benzophenones, 3-hydroxyflavones, and xantones; and those forming O-N-H bridges, such as 2-(2-hydroxyphenyl)benzo-triazoles and 2-(2-hydroxyphenyl)-1,3,5-triazines (commercial examples of these are TINUVIN® and Uvinol® UV absorbers [7]). These molecules exert their UV-absorption activity through the "ESIPT" (Excited State Intramolecular Proton Transfer) mechanism, which involves in the dissipation of incident light energy into non-radiative, low thermal energy due to the presence of intramolecular hydrogen bonds [7]. Another option to overcome the UV light transparency of conventional plastics relies on the melt compounding of metal nanoparticles, i.e., zinc oxide, titanium dioxide, and silver, capable of scattering and absorbing the incident light [8,9]. However, the risk of nanoparticle migration from the package to the product dramatically restricts their usage for food applications as this would not comply with the legislation of food contact materials [10].

In recent years, the interest to alternative molecules of natural origin able to offer adequate UV-absorbing capability has considerably increased. With this regard, polyphenols, including lignin, tannic acids, and flavonoids, as well as melanin, carotenoids, and essential oils have been successfully proven as valid candidates [11].

The incorporation of polyphenols and phenolic acids into polymeric matrices was demonstrated to boost the functional properties of some food packaging materials [12-14]. For instance, gallic acid enhanced both thermal resistance and UV light stability of bio-based high-density polyethylene (bio-HDPE) [15]. In a similar work, adding ferulic acid to poly(lactide) - poly(butylene adipate-co-terephthalate) (PLA-PBAT) blends yielded composite films with improved mechanical (tensile strength), antibacterial, and UV-light barrier properties [16]. Flavonoids, a class of polyphenols, have shown a great potential for different applications in various industries, including as UV-shielding agents in the food packaging sector. Flavonoids have been shown to possess exceptional UV-absorbing capabilities, which can enhance the protection of packaged food from harmful radiation and prevent premature deterioration [17].

In this work we propose the use of natural molecules incorporated into biopolymeric layers coated on PP films to enhance their UV-shielding properties. More specifically, quercetin, curcumin, caffeic acid, and hymecromone (a coumarin derivative) were selected as natural compounds with UV-absorption capability. Coumarin and its derivatives, in particular, are a large family of natural molecules rarely used for food packaging applications, as compared to other natural compounds [18], which justifies our interest toward this class of UV-absorbers. Pectin, a polysaccharide extracted from residues of the food industry, was chosen as the biopolymer for the development of the UV-shielding coating, owing to its good suitability for packaging applications [19,20]. Polypropylene (PP) was selected as the plastic substrate since it is widely used in the packaging sector. Interestingly, PP has poor UV shielding properties and is extremely sensitive to UV light, due to the presence of tertiary protons in the polymeric chain [21,22]. In this study, we first investigated the effect of coating thickness on the UV-shielding behavior of the plastic substrate for each bioactive compound. Then, the combined use of natural molecules was evaluated to possibly reveal any synergistic effect against the transmission of UV light through the substrate. Finally, a comparison with commercial UV-shielding films loaded with UV-barrier molecules of synthetic origin was carried out.

2. Materials and Methods

2.1. Raw Materials and Chemicals

GENU® pectin LM-12 CG, a low ester (degree of esterification ≃ 35%) non amidated pectin extracted from citrus peel, was purchased from CP Kelco (Atlanta, Georgia, USA). Quercetin, curcumin, hymecromone (7-hydroxy-4-methyl-2H-chromen-2-one), and caffeic acid (Figure 1) were purchased from Merck (Milano, Italy). Methanol (VWR, Milano, Italy) was used as the solvent for the

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bioactive compounds. Oriented polypropylene (OPP) 40 µm thick was kindly provided by Vibac spa (L'Aquila, Italy).

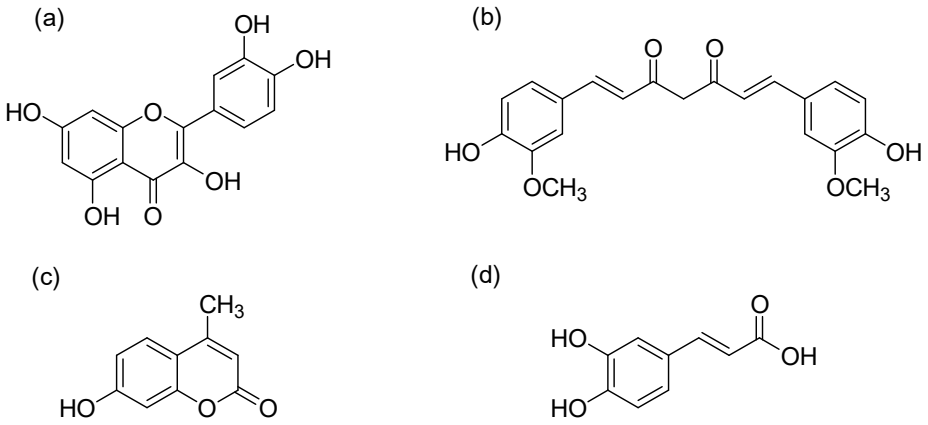

Figure 1. Chemical structure of active compounds used in this study: quercetin (a), curcumin (b), hymecromone (c), and caffeic acid (d).

2.2. Preparation of Coating Solutions and Application to Plastic Films

The pectin-based coatings were prepared according to the procedure depicted in Figure 2.

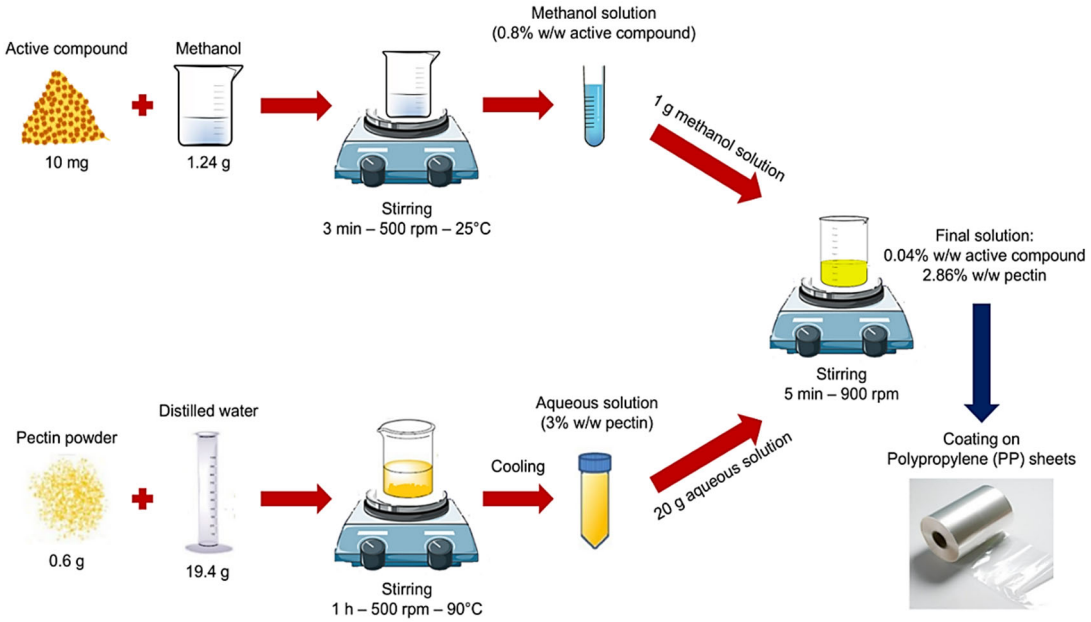

Figure 2. Schematic sketch of the preparation of biopolymer coating solutions.

Specifically, 10 mg of each active compound was solubilized into 1.24 g of methanol under mild stirring to produce 0.8% (w/w) alcoholic solutions. Meanwhile, 0.6 g of pectin powder was slowly dispersed in 19.4 g of distilled water at 90°C until full dissolution. The aqueous solution was then allowed to cool down to 25°C in a water bath and subsequently mixed with the active compound solution at a ratio of 20:1 (g/g), finally obtaining coating solutions with active compound and pectin concentrations of 0.04% and 2.86% (w/w), respectively.

A few mL of the coating solution was applied on the surface of rectangular OPP specimens (24 × 18 cm^2) by means of an automatic film applicator (model 1137, Sheen Instruments, Kingston, UK) at a constant speed of 150 mm min^{-1}, in accordance to ASTM D823-07-Practice C, using steel rods with engraved patterns.

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To assess the effect of coating thickness on the optical properties of coated PP samples, each pectin/active compound solution was applied on the plastic substrate using 4 different bars with increasing diameter, thus attaining final wet layer thicknesses within the range of 4 – 40 µm (Table 1). Water evaporation was achieved by applying a steady and perpendicular flow of mild air (25.0 ± 0.5 °C for 3 minutes) at a distance of 40 cm from the applicator. Regardless of the tested active compound, the theoretical dry thickness shown in Table 1 was calculated based on the dry weight content (2.9 % w/w) of coating solutions.

In a second set of experiments, pectin solutions containing hymecromone (0.02% w/w) and caffeic acid (0.02% w/w) were deposited onto PP strips using bar n°4 yielding a theoretical dry thickness of 1.2 µm. The choice of testing only this combination arose from the best UV-shielding activity shown by the single bioactive compounds when loaded in coating systems (see Results and Discussion section).

Table 1. Wet and dry coating thicknesses as a function of the employed coating bar.

Bar n°	Wire diameter [mm]	Wet coating thickness [µm]	Dry coating thickness [µm]
1	0.05	4	0.12
2	0.15	12	0.36
3	0.30	24	0.72
4	0.51	40	1.2

All coated films were then stored at 25°C in a desiccator before characterization. Uncoated PP films were also analyzed and used as control samples.

2.3. Characterization of Films

The optical properties of coated and uncoated OPP films were analyzed using a high-performance UV-Vis spectrophotometer (Lambda 650, PerkinElmer, Waltham, MA, USA). equipped with a 150 mm diameter integrative sphere, allowing the diffuse transmitted light to be fully captured.

A quantitative assessment of the UV-Vis transmission properties was carried out by recording transmittance spectra between 200 nm and 800 nm. As far as the haze (in %) of coated and uncoated films was concerned, according to the ASTM D1003 standard this parameter was measured in the visible wavelength range (380 - 780 nm). Haze is defined as the percentage of transmitted light that scatters by more than 2.5° from the direction of the incident beam. In practice, haze relates to the ability of a material to clearly display items placed behind it [23,24].

2.4. Statistical Analysis

All experiments and analyses were conducted in triplicate. Data were analyzed using IBM SPSS Statistics 20 software (IBM Corp., Armonk, New York, USA), with a one-way analysis of variance (ANOVA). Tukey's test was used to check for significant differences among samples ($p \leq 0.05$).

3. Results & Discussion

3.1. Effect of Coating Thickness on the Optical Properties of the Plastic Substrate

Figure 3 shows the UV-Vis spectra of OPP, both uncoated and pectin-coated using quercetin, curcumin, hymecromone, and caffeic acid as active compounds. The pectin-coated OPP sample without any active compound was excluded from the analysis, as its UV-Vis spectrum overlapped with that of the uncoated OPP (data not shown). Transmittance values above 80% were recorded for the uncoated OPP within the range of 240 – 800 nm. Irrespective of the employed active compound, a similar performance was displayed by pectin-coated OPP films, but only at wavelengths greater than 400 nm. This confirms that the coating application on OPP did not alter its optical properties in

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the visible range, that is, the clarity. Reversely, a decrease in UV light transmittance was observed for the coated films as compared to the bare substrate in the following order of active compounds: quercetin < curcumin < hymecromone < caffeic acid (Figure 3).

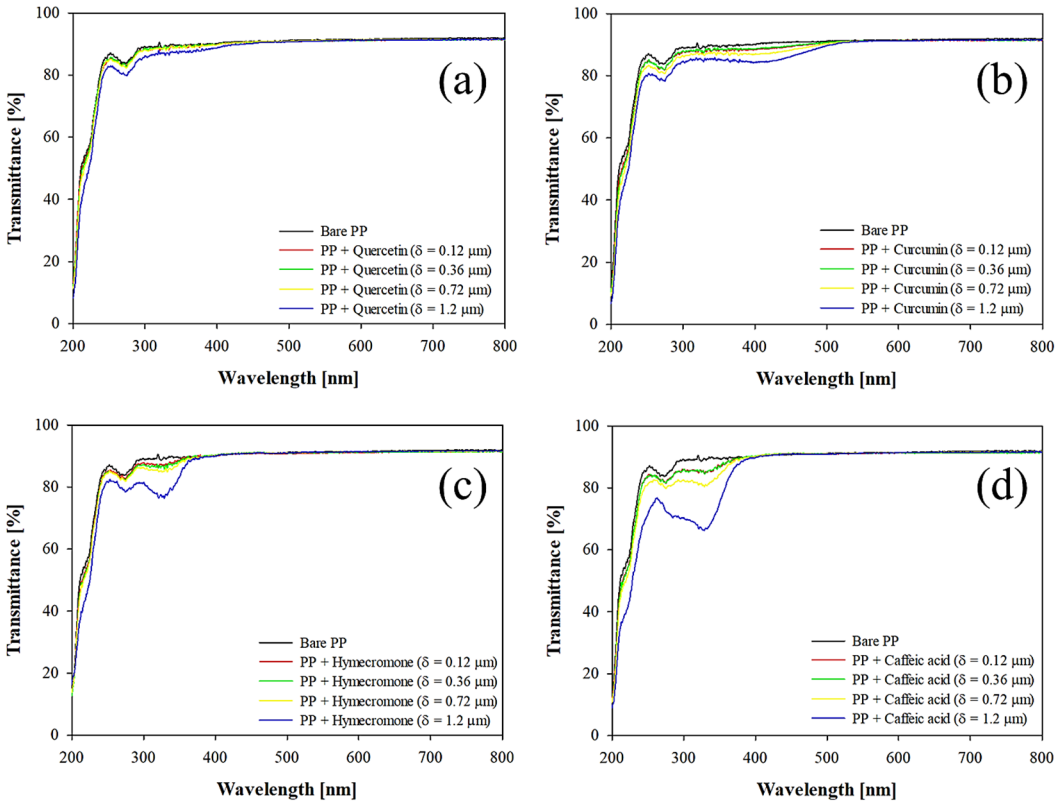

Figure 3. UV-Vis spectra (200 – 800 nm) of uncoated (bare) and pectin-coated OPP in the presence of quercetin (a), curcumin (b), hymecromone (c), or caffeic acid (d) as a function of the coating thickness (δ = 0.12 – 1.2 µm).

The incorporation of hymecromone and caffeic acid in the coating formulations eventually led to the appearance of a saddle-like pattern centered at around 329 nm (Figure 3c-d) typical of hydroxycinnamic acids [25], whose intensity exhibited a thickness-dependent behavior. Indeed, at the highest coating thickness tested in this study (δ = 1.2 µm), minimum transmittance values of 76.4% and 66.4% at λ = 329 nm were measured for pectin-coated films loaded with hymecromone and caffeic acid, respectively (Figure 4).

Most of the UV-induced degradation phenomena in marketed foodstuffs are caused by artificial sources in retail stores emitting predominantly UV-A and UV-B light [26]. Taking this into account, the wavelength of 329 nm was expediently used to retrieve a correlation between transmittance values of coated OPP films and nominal thicknesses of biopolymer coatings in order to predict the thickness of the coating needed to achieve relevant UV-shielding performance for practical purposes.

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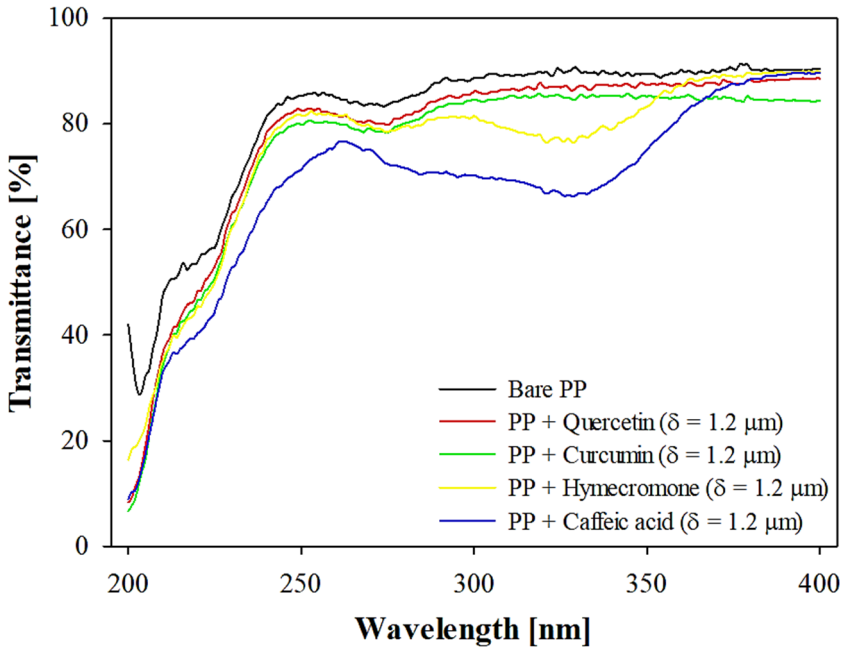

Figure 4. UV spectra (200 – 400 nm) of uncoated (bare) and pectin-coated (δ = 1.2 μm) OPP films using the UV-shielding compounds tested in this work.

To this end, a linear decay model (Eq. 1) was used to fit the experimental data of Figure 3.

$$T_{329nm} = T_0 - Dr_i \cdot \delta \quad (1)$$

Specifically, T_{329nm} (in %) is the transmittance of coated OPP films at a wavelength of 329 nm, T_0 is the transmittance of bare (uncoated) PP films at the same wavelength (89.40%), DR_i stands for the transmittance decay rate (in % μm^{-1}) pertaining to the i-th active compound (i = quercetin, curcumin, hymecromone, and caffeic acid), whereas δ (in μm) is the dry coating thickness. Both experimental (dots) and simulated (dashed lines) data were reported in Figure 5.

The applied mathematical model described the experimental data satisfactorily (average R^2 = 0.92) and unveiled greater DR values when employing hymecromone and caffeic acid as UV-shielding agents (Table 2). It is worth noting that all tested bioactive compounds contain chromophore groups (e.g., aromatic ring, Figure 1) and were used at the same load (10 mg) for coating purposes. The higher molar concentration of hymecromone and caffeic acid, being directly proportional to the absorption by Lambert-Beer law, presumably explains the trend of DR shown in Table 2.

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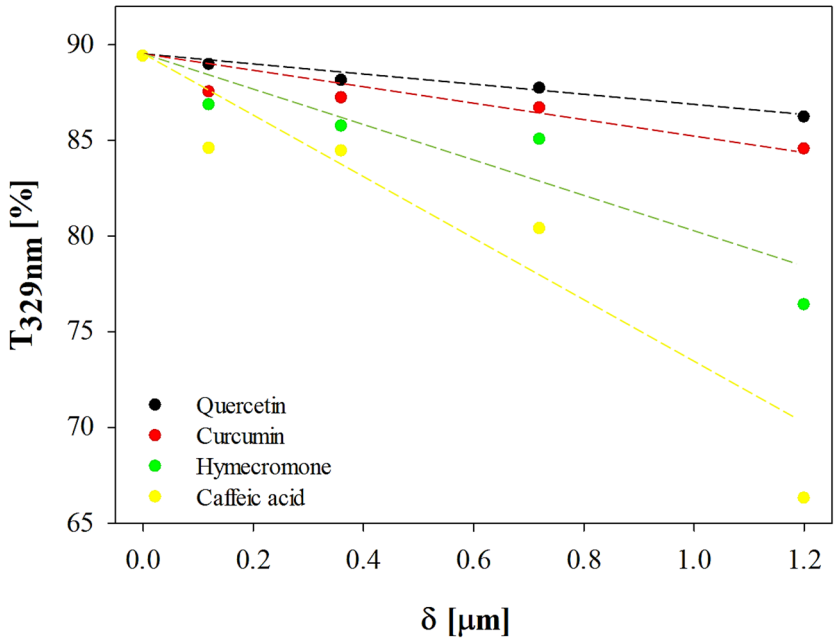

Figure 5. Linear correlations between the dry coating thickness (μm) and the transmittance values of coated PP films (λ = 329 nm) using the UV-shielding compounds tested in this work.

Table 2. Values of T_{329nm} decay rate (DR), obtained from the linear regression of data of Figure 5, and dry thickness of coatings yielding T_{329nm} = 50 % ($\delta_{T329nm=50\%}$) for the UV-shielding compounds tested in this work. The mass ratio between the coating and biopolymer-coated OPP film (CB ratio, in %) is also shown.

Active Compound	DR [% μm^{-1}]	R^2	$\delta_{T329nm=50\%}$ [μm]	CB ratio [%]
Quercetin	2.63	0.98	14.98	27.24
Curcumin	4.17	0.90	9.45	19.11
Hymecromone	9.67	0.90	4.07	9.23
Caffeic acid	18.71	0.92	2.10	4.98

Interestingly, for each active compound, Eq. (1) can be used to estimate the thickness of the dry coating needed to reach target transmittance values for OPP films, for instance, T_{329nm} = 50% (Table 2). According to the linear model, $\delta_{T329nm=50\%}$ for coatings produced with lower-performing molecules (i.e., quercetin, curcumin) are particularly high, thus far exceeding 20% of the thickness of bare OPP film (40 μm). This would unavoidably pose challenges to maintaining the mono-material grade to 95% for full recyclability of the coated film as defined by the EU Regulation 2025/40 [27].

Based on the results collected in Table 2, legislative compliance is only achievable in the case of pectin-coated OPP films loaded with caffeic acid (CB ratio < 5%). Further studies are needed to better understand how the chemical structure of tested active compounds influences the optical properties of OPP-based packaging systems. This would help optimize the loading of active compounds within coating formulations to maximize the UV-shielding performance.

Film clarity is a crucial aspect of food packaging production, as it directly influences consumer acceptance [28]. Haze values of bare PP and pectin-coated OPP samples are embedded in Table 3.

Table 3. Values of haze for uncoated (bare) and pectin-coated OPP films using the UV-shielding compounds tested in this work. The dry thickness of the coating was 1.2 μm. Different superscript letters within the same column indicate statistical differences between samples ($p \leq 0.05$).

Material	Haze [%]
Bare PP	3.90 ± 0.12 ^{ab}
PP + Quercetin	4.26 ± 0.15 ^{b}

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PP + Curcumin	3.78 ± 0.25 [a]
PP + Hymecromone	3.57 ± 0.06 [a]
PP + Caffeic acid	5.40 ± 0.08 [b]

In agreement with previous literature findings [29], the polyolefin substrate displayed a very low haze value. This is ascribable to the limited number of scattering centers within the PP structure, thus curbing the deviation of transmitted light. Coating deposition did not appreciably change the haze of the plastic substrate underneath, even though slight but significant differences ($p \leq 0.05$) were detected between certain active compound pairs, namely curcumin/hymecromone and quercetin/caffeic acid. Overall, none of the tested packaging configurations reached 30% haze, thus indicating no translucency phenomena [30]. The haze degree is determined by surface irregularities that disrupt the uniform distribution of reflected light around the specular angle [31]. Therefore, the homogenous distribution of pectin coating onto bare OPP was supposedly unaffected by the addition of the active compounds investigated in this study. Similarly, applying bionanocomposite coating based on colloidal silica (CS) nanoparticles and pullulan onto bi-oriented PP sheets did not modify the haze thereof [29]. This was explained by the even dispersion of CS nanoparticles in the pullulan network, which then prevented the formation of macro-sized clusters and scattering centers.

The best-performing compounds–hymecromone and caffeic acid–were combined in a 1:1 mass ratio to assess potential synergistic effects. However, no synergy was observed, as confirmed by the overlapping spectra of the compound mixture and caffeic acid alone (Figure 6).

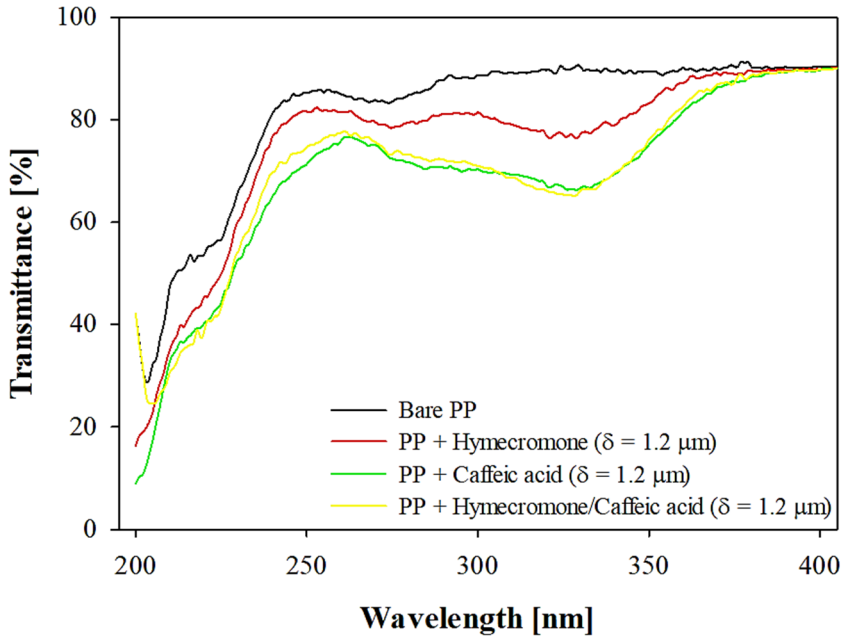

Figure 6. UV spectra (200 - 400 nm) of uncoated and pectin-coated PP films in the presence of hymecromone, caffeic acid, and their combination.

These results seem to indicate that increasing the loading of caffeic acid from 0.02% (w/w) to 0.04% (w/w) apparently did not bring any appreciable improvement in the UV-absorbing feature of the developed packaging films. From a practical point of view, this suggests that increasing the thickness of the coating layer rather than the loading of caffeic acid is a more efficient strategy to improve the UV-shielding property of the plastic film. However, to confirm this hypothesis, additional studies are required.

3.2. Pectin-Coated OPP Films vs. Commercial Solutions

OPP is the preferred material for various food packaging applications due to its unique properties, including high clarity, excellent water vapor barrier properties, and good sealing

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characteristics. However, compared to PET, it generally provides inferior UV-barrier protection, limiting its use in applications requiring UV protection.

To better contextualize the our findings, we compared the performance of the newly developed coated films with commonly used commercial PET-based packaging solutions. Specifically, we used an OPP film coated with a 2.1 µm-thick pectin layer loaded with caffeic acid. For comparison, we selected two commercial materials: (i) the PET top layer of a tray used for fresh pasta (Italian tagliatelle) not incorporating UV-absorbers (commercial solution 1) and (ii) the PET top layer of a tray used for ham packaging, where a UV-shielding material is required to protect the packaged food from detrimental processes, such as discoloration. The results of this comparison are shown in Figure 7.

While no substantial difference was detected between the UV-shielding pectin-coated OPP film and the commercial materials in the visible range, a clear difference concerned the UV region.The packaging solution developed in this study, while showing a remarkable UV-shielding performance compared to the bare OPP substrate, performed decidedly worse than the commercial materials. Both commercial solutions showed a marked cut-off at approximately 380 nm, which is typical of PET and due to the aromatic ring of the main backbone. The same trend has been observed for the OPP film coated with the pectin coating loaded with caffeic acid, which has an aromatic ring too. However, the descending part of the UV-Vis spectum stops abruptly at approximately 330 nm to rise back up to the peak of transmittance centered at 260 nm. Interestingly, the mismatch between the two commercial films is due to the presence of the UV-absorber additive in the commercial film 2.

The additive enhances the UV-shielding capability of the material between 350 nm and 300 nm, which is the wavelength range where most UV-induced degradation phenomena during food storage at manufacturing facilities and in retail stores occur due to both UV-a and UV-b radiation emitted by artificial lamps [26]. Between 200 nm and 300 nm, both commercial films exhibited near-zero absorbance, whereas the OPP-coated film showed significantly higher transmittance values—ranging from ≃ 38% at 300 nm to ≃ 15% at 200 nm, with a peak of ≃ 60% at 280 nm. These results clearly indicate that, although the packaging film developed in this study represents a significant improvement over the bare OPP substrate, it still does not match the UV-absorbing performance of commercially-available materials, thus limiting its potential for widespread application in UV-sensitive foods.

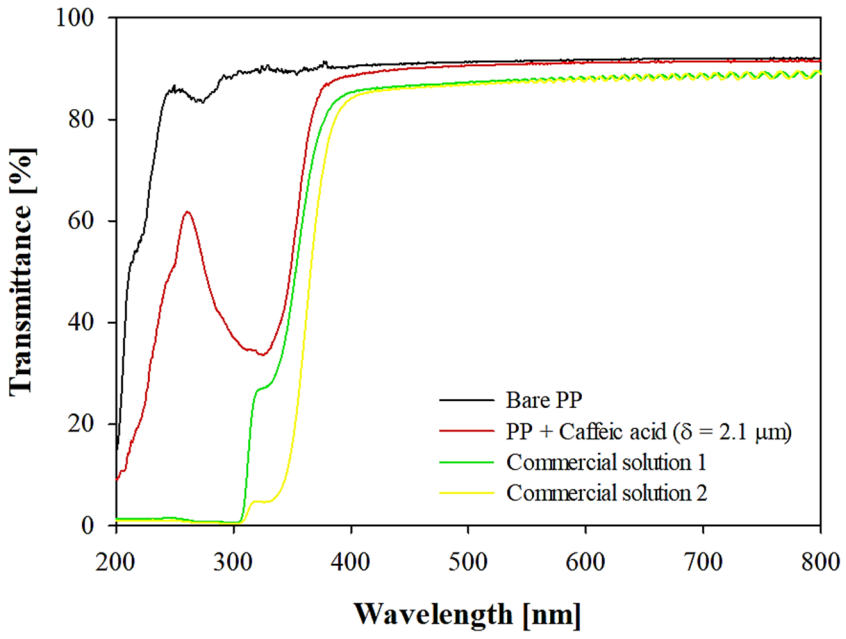

Figure 7. UV-Vis spectra (200 - 800 nm) of uncoated and pectin-coated OPP films containing caffeic acid (δ = 2.1 µm), as well as of two commercial materials endowed with UV-shielding properties.

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4. Conclusions

This study underscored the potential of biopolymer coatings based on pectin and four bioactive compounds to reduce the transmission of UV light when applied on OPP films. The greater UV-shielding effect was measured for caffeic acid-loaded coatings, which showed the highest transmittance decay rate (DR = 18.71 % μm^{-1}) at the target wavelength of 329 nm. In spite of these promising results, the pectin-based coatings with UV-absorbing properties were less performing than commercially-available solutions based on PET incorporated with synthetic UV-absorbing additives. To reduce this gap, future works will focus on exploring the use of other bioactive molecules (e.g., hydroxycinnamic acids and their derivatives) in coating formulations, as well as considering biopolymeric matrices with inherent UV-shielding features.

Author Contributions: For research articles with several authors, a short paragraph specifying their individual contributions must be provided. The following statements should be used "Conceptualization, M.G., D.B. and S.F.; methodology, M.G., D.B. and S.F.; validation, M.G., D.C. and S.F.; formal analysis, M.G. and D.B.; investigation, M.G. and D.B.; resources, A.P., S.D. and S.F.; data curation, M.G., D.B. and D.C.; writing—original draft preparation, M.G. and D.B.; writing—review and editing, D.C., A.P., S.D. and S.F.; visualization, M.G. and D.C.; supervision, S.F.; funding acquisition, S.D. and S.F. All authors have read and agreed to the published version of the manuscript." Please turn to the CRediT taxonomy for the term explanation. Authorship must be limited to those who have contributed substantially to the work reported.

Funding: This study was carried out within the Agritech National Research Center and received funding from the European Union Next-GenerationEU (PIANO NAZIONALE DI RIPRESA E RESILIENZA (PNRR) – MISSIONE 4 COMPONENTE 2, INVESTIMENTO 1.4 – D.D. 1032 17/06/2022, CN00000022). This manuscript reflects only the authors' views and opinions, neither the European Union nor the European Commission can be considered responsible for them.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data are contained within the article.

Acknowledgments: Not applicable.

Conflicts of Interest: The authors declare no conflicts of interest.

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