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Natural Antioxidants as Thermal Stabilizers in Post-Consumer Polypropylene

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ABSTRACT

The upstream applications of post-consumer polypropylene (PCPP) demand long-term oxidation stability to prevent deterioration of its properties. This study explores the potential to replace a commercial antioxidant (AO), Irganox 1010 (I1010), with greener alternatives, quercetin and tocopherol. The emphasis was placed on evaluating their influence on thermal stability, including oxidation resistance and degradation behavior during multiple processing cycles. The rPCPP exhibited inferior thermal properties, with an oxidation induction time (OIT) of 1.2 min and an oxidation onset temperature (OOT) of 205°C. Incorporation of the selected individual AOs improved these properties. The combination of quercetin and tocopherol (QT) exhibited the best results, increasing OIT to 43 min and OOT to 260°C. After five reprocessing cycles, the melt flow rate (MFR) values for the QT system remained unchanged, whereas I1010-stabilized and the non-stabilized rPCPP increased by 183% and 376%, respectively. Similarly, the carbonyl index (CI) value increased by four times for rPCPP and two times for I1010, whereas the QT remained nearly unchanged. The observed enhanced stability of QT is attributed to the synergistic effect. This effect was further supported by the DPPH assay. All the findings demonstrate the effectiveness of selected natural AOs, particularly QT, as a sustainable alternative to conventional stabilizers.

1 | Introduction

In recent decades, multiple factors such as the ever-increasing global plastic demand, the mismatch between production and recycling, societal pressure, governmental and EU legislations [1, 2], depletion of fossil-based resources, possible disruption of supply chains, and economic reasons have culminated in the need to focus on research and development activities, production, and product design to align with sustainable development and a plastic-based circular economy. Despite the suitability of biodegradable plastics for single-use applications, fossil-based polyolefins still remain the most widely used polymers in the packaging sector, owing to their low cost and relatively inexpensive synthesis routes [3]. Among polyolefins, PP is the most

widely used and demanded thermoplastic, representing 16% of plastic demand in Europe [4] because of its unique physical, rheological, mechanical, and chemical properties. Compared with other polyolefins, the presence of a methylene group on every second carbon atom along the polymer chain restricts chain rotation, resulting in reduced flexibility and thus higher stiffness. The arrangement of the methyl group is directly related to its crystallinity. In isotactic PP, all the methyl substituent groups are on the same side of the polymer backbone, giving it a highly regular structure. This regularity allows the chains to pack closely and form an ordered crystalline region, resulting in high crystallinity [5]. But since no real polymer is perfectly isotactic, and chain entanglements prevent complete crystallization, an amorphous fraction always co-exists.

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Polyolefins, in general, and PP in particular, are produced in large quantities due to their extensive use in packaging. Besides that, the U.S. Food and Drug Administration [6], and EU Regulation (EU) No. 10/2011 [7] has approved its use as a safe-for-food-contact material due to its resistance to moisture uptake, inertness to various food types, and its heat-deflection properties that prevent leaching. However, its extensive use contributes significantly to plastic pollution after the end-of-life phase [8]. Plastic recycling represents one of the most effective strategies to mitigate this issue. Among the various recycling approaches, mechanical recycling is considered to be the most practical due to its operational flexibility and economic feasibility [9].

Despite its widespread use, mechanical recycling also has several limitations. A major challenge is the inherent inferior quality of post-consumer recycle (PCR), which depends on the pre-reprocessing conditions, such as thermal, mechanical, and chemical history, as well as exposure to environmental factors. In addition, contamination by non-intentionally added substances, such as dirt, organic residues, and metallic particles, further deteriorates material quality during reprocessing. Moreover, repeated processing degrades the polymer matrix, leading to a decline in thermomechanical performance [10]. The degradation of mechanical properties is linked to chain scission and the generation of free radicals, which occurs during prolonged exposure to conditions such as elevated temperatures, UV radiation, high electric fields, and organic solutions during the previous product life [11, 12].

Antioxidants and UV stabilizers are the most prominent additives that prevent or slow the oxidation of polymers, thereby preventing chain scission, crosslinking, discoloration, and aging. Currently, the most used AOs are phenolic compounds. There are four mechanisms that have been reported in the literature for the stabilization effects of phenolic compounds: single-electron transfer (SET), sequential proton loss electron transfer (SPLET), radical adduct formation (RAT), and hydrogen atom transfer (HAT) [13]. In the case of polyolefins, HAT is the most preferred and accepted stabilization mechanism.

PP degrades via a chain-scission mechanism, where thermal or UV exposure generates free radicals that react with oxygen to form peroxy radicals and hydroperoxides [14]. The decomposition of these species produces additional radicals that accelerate degradation, while AOs terminate this process by scavenging free radicals [15, 16]. Currently, synthetic phenolic AOs such as Irganox 1010 and Irganox 1076 are used as primary AOs (also called chain-breaking AOs), and their role is to prevent the formation of free radicals by donating hydrogen atoms to neutralize them, whereas secondary AOs (also known as hydroperoxide decomposers), such as phosphites and thioesters, decompose hydroperoxides [17].

However, recent studies have revealed that these synthetic AOs, or their byproducts, can adversely affect organisms and even trigger environmental effects, sparking great public concern about environmental issues and potential impacts. Since synthetic AOs are not covalently bonded to the polymeric materials, they can be readily released from the polymers into the environment during production, use, recycling, and disposal [18, 19].

Many studies have reported pollution and associated toxic effects caused by low-molecular-weight synthetic phenolic AOs [20, 21]. For example, 2,6-di-tert-butyl-hydroxytoluene (BHT) has been broadly detected in various environmental media (air, dust, sediment, water, soil, and sludge) and the human body (blood, breast, urine, milk, and finger nails) [22]. Pack et al. identified nine potential migrants that have certain environmental risks, including the plastic additives and non-intentionally added substances from food packaging and disposable containers made of polyethylene, polypropylene, and polystyrene [23]. Due to the growing environmental concerns, especially in food-contact plastic packaging, the search for new additives for polymers has been ongoing for several years.

In response to these concerns, there has been growing interest in developing safer, more sustainable additive systems. One promising approach is to use green or bio-based alternatives, such as natural AOs, to replace synthetic AOs. A wide range of natural plant-derived AOs, including leaves, fruits, and roots, have been investigated [24]. These compounds offer key advantages, including low toxicity, biodegradability, and reduced environmental impact, making them suitable for applications in food packaging and biomedical fields. Recent studies demonstrated that silymarin extracts [25], natural oils [24], carotene [26, 27], curcumin [28, 29], lignin [30, 31], flavonoids [31], and vitamin E (α -tocopherol) [32–35], in enhancing melt stabilization and removing thermo-oxidative resistance in polymers.

One of the most prominent natural AOs is α -tocopherol, also known as vitamin E. Structurally, it consists of a mixture of soluble phenolic compounds, comprising a chromanol ring and a 16-carbon phytyl chain. It acts through a chain-breaking mechanism, yielding excellent results, even at low concentrations (0.1%–2%). It also prevents a greater reduction in the MFR of LDPE, even after repeated extrusions [34]. The reaction of α -tocopherol with alkyl peroxy radical leads to the formation of tocopheroxyl radical, which isomerizes to benzyl radical. This radical is involved in various other reactions that lead to the formation of spirodimers, dihydroxydimers, and trimers [32, 34]. Most of these by-products themselves possess significant stabilization effects, which were confirmed by Al-Malaika's study on the melt stabilization of PP [33].

Quercetin is regarded as one of the most abundant flavonoids obtained from natural resources. It is found in tea, vegetables, fruits, and wines. The polyphenol quercetin has been the subject of multidisciplinary research due to its antioxidative, anti-inflammatory, anticarcinogenic, and antiviral properties, as well as its ability to act as a free-radical scavenger and a metal-ion chelator [36–38]. Quercetin contains two benzene rings connected by a pyrene ring [39]. It comprises five hydroxyl groups in its structure, which contribute to its biological properties [37]. Samper et al. studied the stabilization of PP against thermo-oxidation and UV behavior using five naturally derived polyphenols from flavonoid structures: quercetin, chrysin, hesperidin, naringin, and silibinin. The results showed that the flavonoid compounds quercetin and silibinin provided excellent protection against oxidation and UV radiation [40]. Yang et al. investigated the effects of quercetin on the properties of poly(butylene adipate-co-terephthalate)

(PBAT)/thermoplastic starch films. The films demonstrated excellent antioxidant activity and UV protection, owing to the incorporation of quercetin [41].

The stabilization of recycled PP using natural AOs has attracted increasing attention as a sustainable alternative to conventional additive systems. This study provides an in-depth analysis of the potential use of quercetin and α -tocopherol as natural AOs in rPCPP. While quercetin and tocopherol are recognized individually for their antioxidant activity. However, literature scarcely addresses their use in complex, heterogeneous materials such as rPCPP, which may contain pre-existing free radicals and pro-oxidant compounds. Moreover, they may pose unique processing challenges due to their physical, chemical, and thermomechanical history. This study further demonstrates that oxidation-resistant rPCPP with enhanced thermal stability can be developed by exploiting the synergistic relationship between the tocopherol and quercetin. To our knowledge, this synergistic combination has not been exploited previously for rPCPP.

2 | Materials and Methodology

2.1 | Materials

Recycled post-consumer polypropylene pellets were kindly supplied by Emballator AB, Sweden. Alpha tocopherol, quercetin (95%), pentaerythritol tetrakis (3,5-di-tert-butyl-4-hydroxyhydrocinnamate)/(Irganox 1010), 2,2-diphenyl-1-picrylhydrazyl (DPPH), absolute ethanol, and dichloromethane were purchased from Sigma Aldrich, Germany.

2.2 | Compounding and Reprocessing

The tocopherol was dissolved in 50 mL of dichloromethane, and the solution was mixed with rPCPP pellets. The assembly was then kept in a vacuum oven at 50°C for 4 h. All other AO combinations were combined with dried PP pellets during the melt compounding. The screws were maintained at 75 rpm, and the heating zones were set to 200°C. The processing conditions for all AO combinations were the same. The compositions of the PP and the AOs are listed in Table 1. The AOs were compounded for 3–4 min in a twin screw 15 cc micro-compounder from Xplore Instruments BV, the Netherlands. Reprocessing of the PCPP and AO formulations was performed by repeatedly extruding the initially prepared AO–PP blends. The materials were first extruded into filaments and

subsequently pelletized. The obtained pellets were then re-processed through the compounder for up to five consecutive extrusion cycles. All processing steps were conducted under the same temperature settings and residence time conditions as described above. The selected natural AOs, along with their main characteristics, are listed in Table 2.

2.3 | Characterization

2.3.1 | Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) was performed using a Q2000 DSC (TA Instruments). Samples weighing 10 ± 1 mg were subjected to a heating cycle from 20°C to 200°C at a rate of 10°C/min under a nitrogen atmosphere with a flow rate of 50 mL/min, followed by cooling to 20°C and a second heating to 200°C under identical conditions. The melting temperature and enthalpy of fusion were determined using Universal Thermal Analysis software. Based on DSC curves, rPCPP was found to contain a PE fraction. The approximate weight fraction of PP is quantified using the Shimadzu method (Equation 1) [42, 43]. The mass fraction crystallinity was calculated using Equation (2).

$$\% \text{ PP} = 100 \times \frac{\frac{\Delta H_{m,\text{samplePP}}}{\Delta H_{m,\text{neatPP}}}}{m_{\text{sample}}} \quad (1)$$

$$X_{c,\text{PP}}(\%) = \frac{\Delta H_{m,\text{samplePP}}}{\% \text{ PP} \times \Delta H_{100,\text{PP}}} \quad (2)$$

where $\Delta H_{m,\text{samplePP}}$ is the measured enthalpy of fusion from the sample, $\Delta H_{m,\text{neatPP}}$ is the enthalpy of corresponding neat PP, and $\Delta H_{100,\text{PP}}$ is the enthalpy of fusion for 100% crystalline PP, taken as 207 J/g. All measurements were conducted in triplicate to ensure reproducibility.

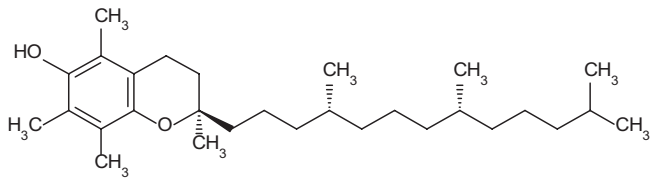
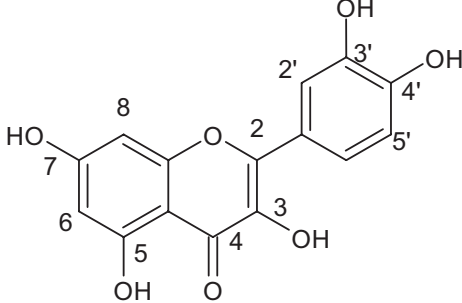
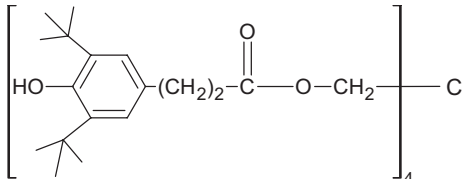
2.3.2 | Oxidation Onset Temperature

OOT tests were performed on a DSC Q2000 differential scanning calorimeter from TA Instruments. To evaluate antioxidant efficiency, 10 ± 1 mg of sample was heated in an aluminum pan from 30°C to 300°C at a heating rate of 20°C/min in an oxygen atmosphere with a flow rate of 50 mL/min. During sample preparation and loading, specimens were handled with gloves to prevent contamination. The onset temperature of this reaction (OOT) was determined by drawing

TABLE 1 | Selected concentrations of AOs in rPCPP.

Sample abbreviations	Recycled PCPP (wt.%)	Tocopherol (wt.%)	Quercetin (wt.%)	Irganox 1010 (wt.%)
rPCPP	100	—	—	—
Q	99.75	—	0.25	—
T	99.75	0.25	—	—
I1010	99.75	—	—	0.25
QT	99.75	0.125	0.125	—

TABLE 2 | Selected AOs, their structure and properties.

Sample	Structure	Molecular weight (g/mol)	Melting point (°C)
Tocopherol		430.71	3.5
Quercetin		302.24	316
Irganox 1010		1177.63	117

a tangent to the exothermic curve, which follows after the endothermic melting peak. Three replicates were tested for each type to ensure reproducibility.

2.3.3 | Oxidation Induction Time

Oxidation Induction Time (OIT) for different samples was measured using the same TA Instruments DSC Q2000 device. Approximately 10 ± 1 mg of the sample was placed in an aluminum pan and heated to 200°C at a rate of $20^\circ\text{C}/\text{min}$ under a nitrogen purge stream ($50 \text{ mL}/\text{min}$). Then, the material was held at that temperature for 5 min under isothermal conditions. After 5 min, the gas purge flow was switched to pure oxygen. Samples were handled with gloves to avoid contamination. OIT was determined by drawing tangents at the point of inflection on the DSC curve. Three replicates were tested for each type.

2.3.4 | Thermogravimetric Analysis

The efficiency of AOs was evaluated by using a thermogravimetric Analysis (TGA) Q500 thermogravimetric analyzer instrument from TA Instruments. Approximately 9–11 mg of each sample was weighed into cleaned platinum pans. The experiments were conducted in dynamic heating mode at a heating rate of $10^\circ\text{C}/\text{min}$ in a nitrogen atmosphere from 25°C to 700°C . The same conditions were used for all the samples. Three

replicates were tested. The analysis was performed on the TA Universal Analysis 2000 software.

2.3.5 | Melt Flow Rate

The melt flow rates (MFR) of the samples ($\text{g}/10 \text{ min}$) were measured at 200°C and 2.16 kg load using a Tinius Olsen MP 1200 machine. A minimum of five measurements was taken for each sample type, and the average MFR was calculated.

2.3.6 | Fourier Transform Infrared Spectroscopy

Fourier Transform Infrared Spectroscopy (FTIR) was used to analyze the oxidative degradation after multiple processing cycles. The FTIR spectra for all samples were obtained at room temperature using an ATR-FTIR spectrophotometer (Nicolet iS10, Thermo Scientific, USA). The samples were scanned in the IR range of $4000\text{--}400 \text{ cm}^{-1}$ wavenumber with a total of 32 scans. The ATR crystal was diamond, and the penetration depth of the evanescent wave was $2.3 \mu\text{m}$. A minimum of three replicates was tested for each sample. Finally, the carbonyl index values were calculated using the following formula:

$$\text{carbonyl index (CI)} = \frac{A_c}{A_R} \quad (3)$$

where A_c is the area of the carbonyl absorption band (1820–1620 cm^{-1}), and A_R is the area of the reference band (1400–1500 cm^{-1}).

2.3.7 | Antioxidant Activity

2.3.7.1 | DPPH assay. The spectrometric assay uses 2,2-diphenylpicrylhydrazyl (DPPH) radical as a reagent. One milliliter of the AO samples with various concentrations in ethanol was added to 3 mL of 0.2 mM DPPH dissolved in ethanol [44]. The solution was incubated for 30 min, and then the absorbance was measured against the blank at 517 nm using the Genesys 180 UV-Vis spectrophotometer from Thermo Scientific. The radical scavenging activity (RSA) was determined by using Equation (4). The AO concentration that provided 50% of RSA was determined graphically.

$$\% \text{RSA} = \frac{A_{\text{blank}} - A_{\text{sample}}}{A_{\text{blank}}} \times 100 \quad (4)$$

3 | Results

3.1 | Thermal Analysis

DSC analysis, shown in Figure 1, reveals that the rPCPP exhibits two melt peaks: one slightly feeble at 126°C and the other at 163°C. This indicates that rPCPP is a two-phase mixture of PP and PE, with melting temperatures as described above. The PE composition in the blend was determined using the method described in Section 2.3.1. The estimation suggests that the blend contains approximately 3.4% PE. The presence of PE in PP is more likely to be linked with the sorting efficiency at the material recovery facility. Even near-infrared is not flawless, and some discrepancies may occur due to multilayered components, pigments, contaminated surfaces, or similar densities [45]. Additionally, some recyclers intentionally add PE as a comonomer to impart specific properties, such as melt stability, toughness, and oxidation resistance [46].

The values of OIT and OOT are shown in Table 3. The data indicate that rPCPP exhibits inferior thermal stability. The analysis reveals that all AOs employed in the study enhance the thermal properties of rPCPP. However, some AOs performed

better than others. The comparison of the individual AOs reveals that quercetin performed better than α -tocopherol and I1010. This enhanced stability against degradation is due to the flavonoid structure, which features multiple OH groups in the phenolic rings. Phenolic compounds act as radical scavengers. When free radicals are generated by thermal oxidation, phenolic compounds neutralize them or slow their reactions, thereby hindering molecular scission. A further improvement in the antioxidant ability of quercetin was observed when α -tocopherol was added to quercetin in a 1:1 by weight ratio. This improvement in antioxidant ability was linked to the synergistic effect of the two AOs [47]. This combination gives the highest OIT value of 43.5 min. OIT measurements provide reliable information on the material's thermal stability. Some polymers, such as polyolefins, do not immediately oxidize when exposed to oxygen under isothermal conditions until a certain amount of time has passed, known as the induction period. The longer the induction time, the better the thermal stability, the longer the service life, and vice versa. However, it does not replicate the processing conditions under which the plastic is exposed to high temperatures and oxidation. Therefore, the OOT was determined. OOT is widely used to analyze the thermal oxidative stability of polymers in both academic and industrial settings, most preferably to assess the stability of polyolefins [48]. The OOT analysis yielded a similar pattern to that of OIT, as presented in Table 3. In the case of non-stabilized PP, OOT was observed at 205°C. The OOT values suggest that both tocopherol and I1010 have similar potentials, whereas quercetin outperformed the aforementioned AOs. The highest OOT was observed in the sample QT. A plausible reason for this could be the synergistic effect of quercetin

TABLE 3 | OIT and OOT values extracted from DSC thermograms.

Sample	OIT at 200°C (min)	OOT (°C)
rPCPP	1.2 ± 0.05	205 ± 1
Q	15.6 ± 0.4	244 ± 2
T	12 ± 0.2	240 ± 0.2
QT	43.5 ± 1.0	260 ± 2
I1010	12.6 ± 2.0	236 ± 3

Note: All time values are reported in minutes (decimal format).

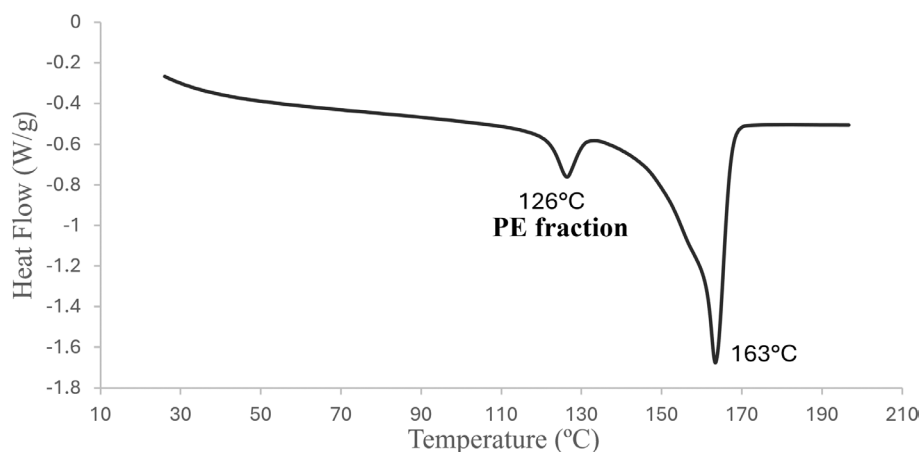


FIGURE 1 | DSC thermogram of rPCPP with PE fraction.

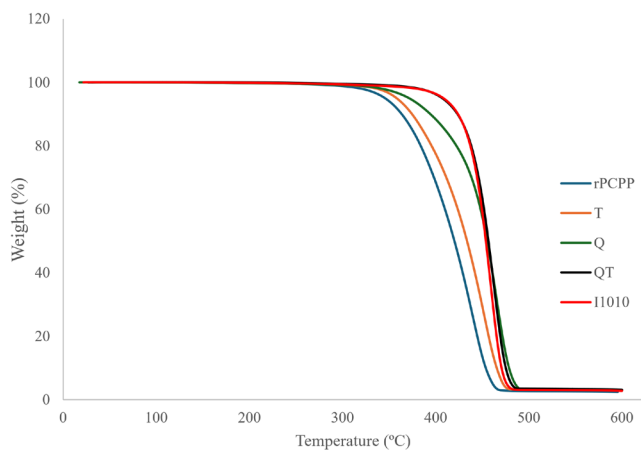


FIGURE 2 | The TGA curves showing the effect of AOs on thermal properties of stabilized and non-stabilized samples. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com)]

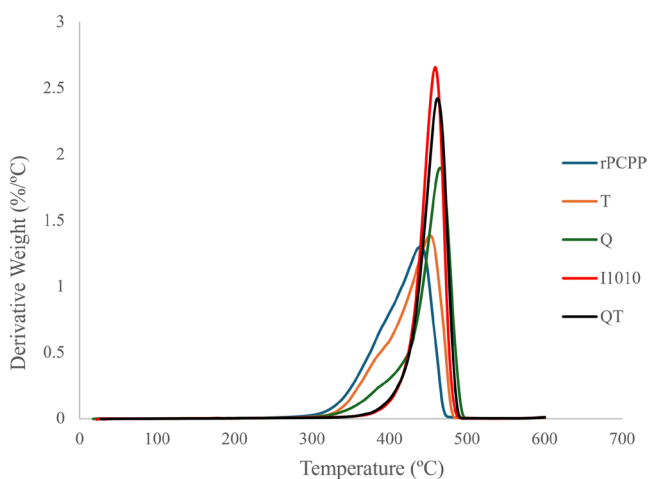


FIGURE 3 | The DTG curves derived from TGA curves. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com)]

TABLE 4 | The onset degradation temperature (T_{ons}), 10% weight loss ($T_{10\%}$), and maximum degradation temperature (T_{max}), derived from TGA and DTG curves.

Samples	T_{ons} (°C)	$T_{10\%}$ (°C)	T_{max} (°C)
rPCPP	383	363	438
T	398	374	452
I1010	435	424	460
Q	429	394	465
QT	436	423	462

and tocopherol, which may be attributed to either their protective effects or to their repair and regeneration mechanisms [49, 50]. Both effects are explained in the next section.

Figures 2 and 3 show TGA and derivative thermogravimetric (DTG) analysis, respectively, for the samples. Some important parameters, such as the onset degradation temperature (T_{ons}), 10% weight loss ($T_{10\%}$), and maximum degradation temperatures

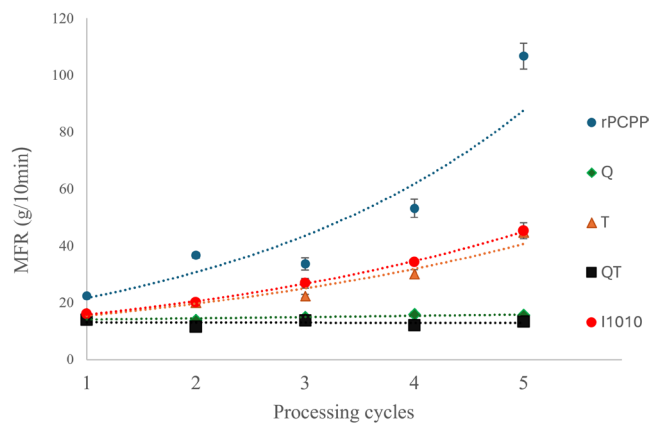


FIGURE 4 | The effect of reprocessing on MFR of stabilized and non-stabilized samples. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com)]

(T_{max}), are presented in Table 4. It is essential to know the thermal stability in aggressive environments. Such conditions artificially accelerate degradation and provide rapid diagnostic tools for determining the relative stability of materials that, otherwise, in working environments, are too tedious to assess. As expected, the rPCPP shows lower values than the stabilized samples. This is because all the AOs are phenolics. A phenolic AO contains an OH functional group, which can donate hydrogen atoms to neutralize active free radicals and form stable products, thereby preventing or delaying degradation. One can observe that the samples containing quercetin and the quercetin–tocopherol combination exhibit a higher stability, comparable to that of the commercial AO I1010. The OIT and OOT results were also complementary with the TGA results, indicating that the samples containing quercetin show promising oxidative stability.

3.2 | Thermo-Mechanical Degradation During Reprocessing

Thermomechanical degradation is one of the inherent limitations of the mechanical recycling process. Therefore, the effects of degradation were evaluated by examining variations in MFR with the number of processing cycles (Figure 4). Since MFR relates to the rheological properties of the material and is often linked with the molecular weight. Thus, potential changes in molecular weight due to oxidative degradation during reprocessing can be indicated by changes in rheological properties measured by MFR. The results revealed that rPCPP shows an increase in MFR values after each recycling step except the third cycle, which is within error. This decrease in MFR may be attributed to the branching and cross-linking of PE molecules, as reported by other studies [51–53]. At the end of the fifth reprocessing cycle, the MFR exceeded 100 g/10 min. This result implies that the polymer chains of rPCPP undergo chain scission. This behavior results from thermomechanical degradation during reprocessing, as the reduction in molecular weight lowers viscosity, thereby increasing the MFR [54]. Unlike rPCPP, the tocopherol-modified polymer maintained stability in MFR for up to 3 cycles, with a rapid increase, indicating that AOs were consumed after the third cycle. A similar pattern is also observed with I1010. On the other hand, the QT combination

showed remarkable resistance to thermo-oxidative degradation and even exhibited lower MFR values after second reprocessing. This may be attributed to the synergistic effect of two AOs. Another reason could be better homogenization of quercetin after the second cycle, since PP is a non-polar polymer, whereas quercetin is polar, which could lead to poor solubility

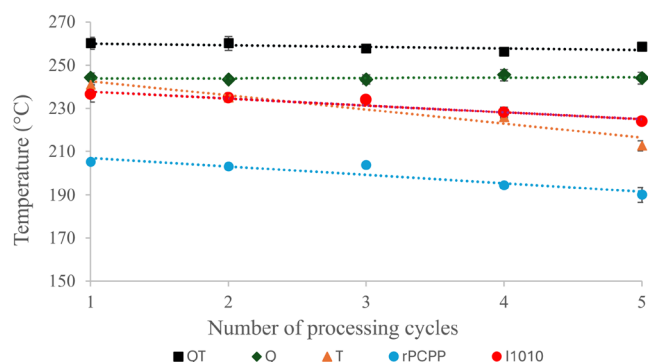


FIGURE 5 | The effect of reprocessing on OOT values of stabilized and non-stabilized samples. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com)]

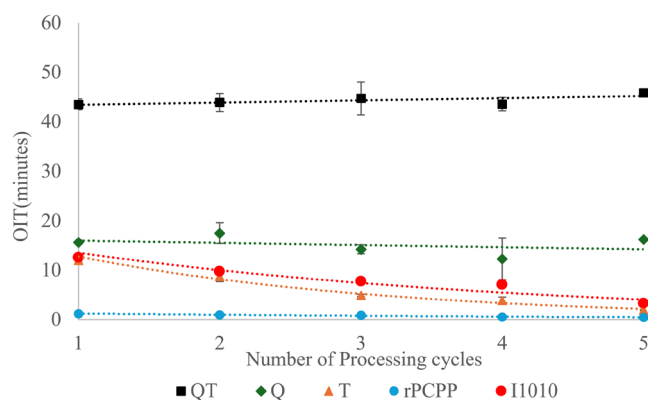


FIGURE 6 | The effect of reprocessing on OIT values at 200 °C of stabilized and non-stabilized samples. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com)]

and phase separation [31]. However, subsequent reprocessing cycles lead to its better homogenization, thereby unlocking its full potential.

The residual stability of each formulation after multiple reprocessing cycles was evaluated by OIT (at 200 °C) and OOT analysis, as shown in Figures 5 and 6, which corroborate the MFR results. Their analysis reveals that the rPCPP shows a continuous decline in OOT and OIT values. The synthetic AO I1010 and tocopherol also show a gradual decline in their ability to preserve OOT properties with successive reprocessing cycles. The OOT value remains stable in samples containing quercetin, even after five cycles. The enhanced stability of quercetin-filled PP, even after multiple reprocessing cycles, was related to the limited solubility of quercetin in PP. During successive processing, the resupply of active molecules and improved homogenization result in greater stability [24, 31]. The combination of tocopherol and quercetin showed an OIT time of 45 min even after five cycles of reprocessing.

Table 5 summarizes the crystallinity (X_c) and melting temperature (T_m) of the samples as a function of processing cycles, obtained from the second heating scan. A decrease in T_m was noted for rPCPP after five cycles, from 163 °C to 161 °C, which can be attributed to thermomechanical degradation of PP. Apart from this reduction, the melting temperatures of other samples remained largely unchanged. This observation is consistent with previous studies, which reported that T_m is unaffected by repeated processing [43, 55], indicating that the crystalline phase during reprocessing was preserved while the amorphous phase was more affected.

Regarding crystallinity, no consistent trend was identified. Instead, minor fluctuations were observed across all the samples, without the systematic change often reported in other studies on PP materials [56]. Degradation of PP typically initiates at the tertiary carbon atom along the polymer chain, leading to chain scission and the formation of low-molecular-weight compounds, such as oligomers and other volatile substances. This process generally results in a broader molecular weight distribution, which can hinder chain folding and reduce

TABLE 5 | The effects of multiple reprocessing on melting temperature (T_m) and crystallinity (X_c) of stabilized and non-stabilized samples.

Sample	Processing cycles	1	2	3	4	5
rPCPP	T_m (°C)	163.3 ± 0.2	163.5 ± 0.7	162.6 ± 0.1	161.6 ± 0.1	161.5 ± 0.1
	X_c (%)	32.6 ± 0.5	32.2 ± 2.2	34 ± 0.3	31.7 ± 1.7	32.9 ± 2
Q	T_m (°C)	164.1 ± 0.7	163.6 ± 0.3	163.4 ± 0.1	164.3 ± 1.3	163.8 ± 0.5
	X_c (%)	30.6 ± 1.1	32.7 ± 2.7	33.2 ± 0.9	32.2 ± 1.1	32.6 ± 1
T	T_m (°C)	163.2 ± 0.2	163.5 ± 0.7	162.9 ± 0.1	162.8 ± 0.05	162.6 ± 0.2
	X_c (%)	34.7 ± 2.7	34 ± 2.5	33.6 ± 0.3	33.7 ± 1.3	33.9 ± 0.7
QT	T_m (°C)	163.9 ± 0.6	163.9 ± 0.1	163.5 ± 0.2	163.5 ± 0.1	163.3 ± 0.1
	X_c (%)	32.9 ± 1.1	33.3 ± 0.5	34.3 ± 0.9	33.8 ± 0.5	32.8 ± 0.8
I	T_m (°C)	163.4 ± 0.2	163.3 ± 0.1	162.9 ± 0.1	163 ± 0.2	163 ± 0.3
	X_c (%)	32.4 ± 1.3	31.2 ± 0.3	33.2 ± 0.1	32.7 ± 0.5	31.6 ± 0.03

the overall crystallinity [43, 57]. However, the presented results do not follow this expected behavior, as the crystallinity values remained relatively stable throughout the cycles. This suggests that degradation may have been limited or effectively counteracted by the presence of stabilizers or other impurities from the use phase. The absence of a clear trend in crystallinity may also be attributed to competing phenomena, such as the rearrangement and recrystallization of shorter chains formed during reprocessing. Furthermore, this behavior in crystallinity was not entirely consistent with the MFR, OIT, and OOT values, which provide a critical indication of thermomechanical degradation in PCPP.

3.3 | The FTIR Spectroscopy

FTIR spectroscopy was used to analyze the thermal degradation behavior of the samples. Figure 7a,b shows the FTIR spectra (1900–1500 cm^{-1}) of the first and fifth reprocessing cycles, respectively. To analyze degradation across different processing cycles, the progressive broadening of the carbonyl band was analyzed, and carbonyl index (CI) values were calculated and plotted in Figure 8. The carbonyl band results from the overlapping of absorption bands associated with various functional groups, such as ketones, carboxylic acids, aldehydes, and esters [58]. The intensity of the peaks in the

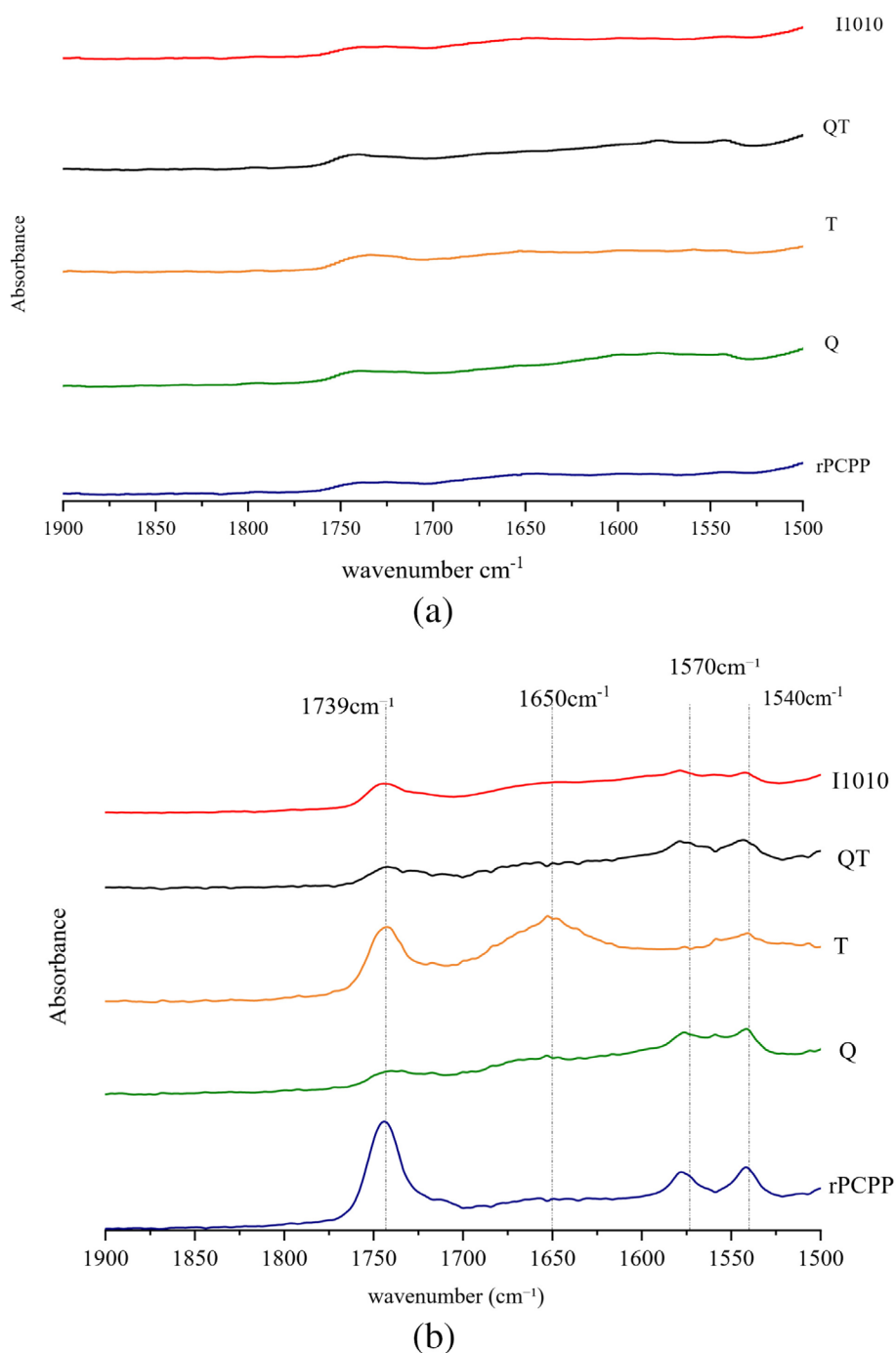


FIGURE 7 | (a) The FTIR spectra (1900–1500 cm^{-1}) of samples from the 1st cycle. (b) The FTIR spectra of samples (1900–1500 cm^{-1}) from the 5th cycle. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com)]

carbonyl region ($1620\text{--}1820\text{ cm}^{-1}$) increases during subsequent reprocessing. The peaks formed at 1739 cm^{-1} are attributed to the ester carbonyl stretching, and the peak at 1650 cm^{-1} is related to the unsaturated carbonyl stretching. Based on the plotted CI values, the non-stabilized PP exhibited the highest overall CI growth across the reprocessing intervals, indicating pronounced susceptibility to oxidative degradation. In contrast, all AO formulations showed a reduction in CI relative to rPCPP, confirming that each AO system suppressed carbonyl-containing oxidation product formation to varying degrees. I1010 exhibited a steady increase in CI values, indicating moderate stabilization. Among the individual systems, quercetin shows lower CI values, indicating greater stability.

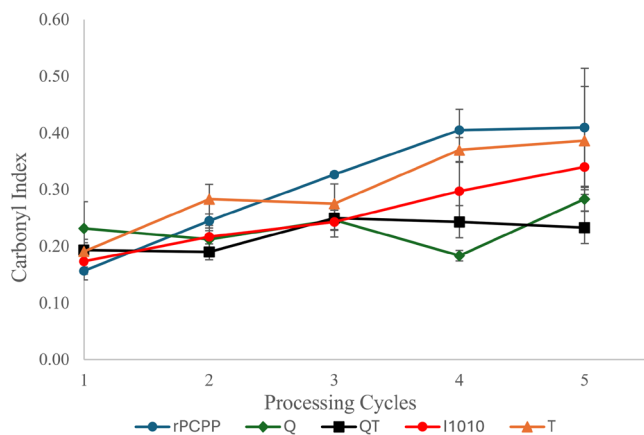


FIGURE 8 | The effect of reprocessing on CI of stabilized and non-stabilized samples. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com)]

Notably, the combined QT system showed greater oxidative resistance than the individual AOs. This indicates a strong synergistic effect between quercetin and tocopherol, resulting in the most effective inhibition of oxidation and the lowest formation of degradation products. In addition, a dual peak at 1540 and 1570 cm^{-1} was observed in rPCPP samples after reprocessing. This may be due to metal-carboxylate formation, specifically the asymmetric and symmetric stretching of the COO^- group [59]. These may have arisen due to carboxylic acids formed during degradation reacting with metal ions (from recycled impurities, catalyst or additive residues, or processing equipment). A possible explanation for the zig-zag pattern observed in Figure 10 is due to the evaporation of low-molecular-weight carbonyl-containing degradation products into the gaseous phase during reprocessing, rendering them undetectable by FTIR analysis [60].

3.4 | DPPH Assay

The relative effectiveness of AOs can be evaluated using the 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging assay, which measures AOs' ability to scavenge the DPPH free radical

TABLE 6 | The inhibitory concentration at 50% RSA derived from DPPH analysis.

Samples	IC ₅₀ ($\mu\text{g/mL}$)
I1010	346
T	120.3
Q	56.6
QT	48.8

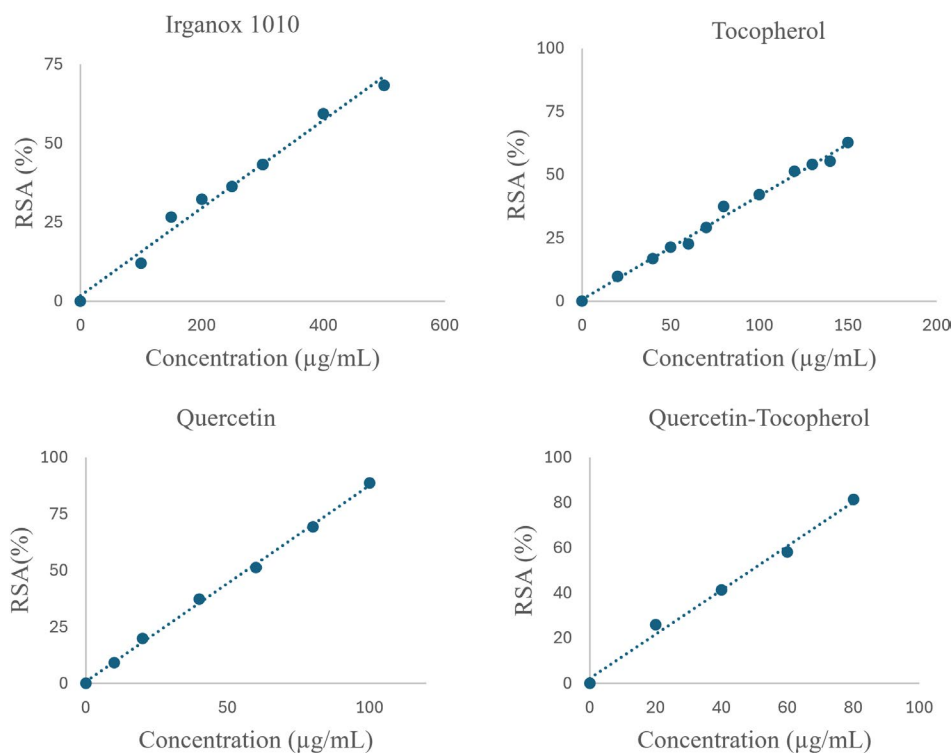


FIGURE 9 | Antioxidant activity of different antioxidants by DPPH assay. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com)]

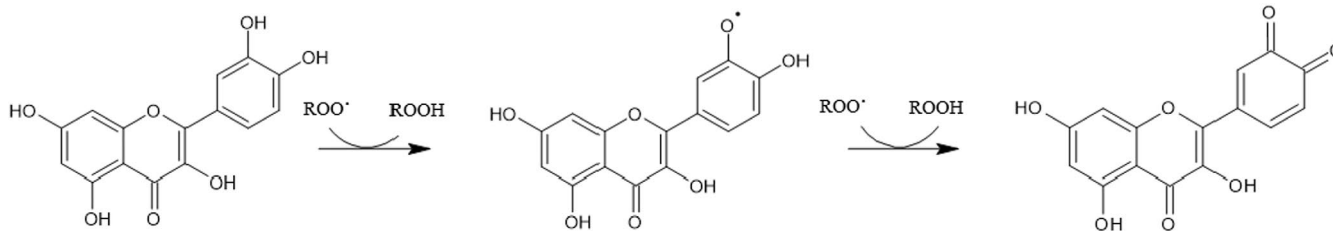


FIGURE 10 | Proposed quercetin antioxidation mechanism.

by donating a hydrogen atom. DPPH has a distinctive purple color and exhibits an absorption maximum in solution at around 517 nm. However, when an AO is added to DPPH, its characteristic purple color gradually fades, progressing from purple to yellow, indicating the radical scavenging activity (RSA). AOs with higher activity require lower concentrations, and color fading occurs more quickly. The absorbance at varying concentrations was measured using a UV-Vis spectrophotometer, and the concentration at which 50% of the RSA is determined graphically, as shown in Figure 9. The inhibitory concentrations at 50% (IC₅₀) of RSA are listed in Table 6. The data indicate that natural AOs exhibit significantly greater antioxidant activity than the conventional phenolic stabilizer I1010. Quercetin and the QT combination are approximately six times more effective than I1010 on a mass basis. One possible reason for the reduced RSA of I1010 is related to its larger molecular size. The larger the molecular size, the fewer OH groups contribute, and hence its ability to react with DPPH is lower, thereby lowering its scavenging activity [61]. The lowest IC₅₀ value in the QT system underlines the synergistic effect between quercetin and tocopherol, enabling the combined system to scavenge DPPH radicals more efficiently than individual AOs. The enhanced antioxidant activity of the AO mixture is consistent with its superior performance, as observed in the complementary DSC and MFR analyses.

4 | Discussion

All the natural AOs in this study are phenolic compounds that act as radical scavengers. The free radical scavenging activity of phenolic AOs (Ar-OH) is characterized by their ability to donate hydrogen atoms and thereby scavenge radicals [62]. They neutralize free radicals by donating hydrogen atoms, forming relatively stable phenoxyl radicals (ArO·), and breaking the chain oxidation reaction, as shown in Equation (5).



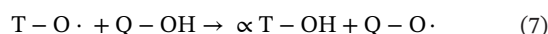
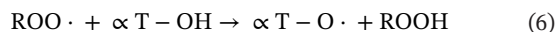
Quercetin shows excellent AO properties, owing to multiple hydroxyl groups in the quercetin molecule. Additionally, quercetin exhibits high reactivity due to the presence of the OH groups in the carbon ring [63]. Quercetin functions as a scavenger by rapidly donating a hydrogen atom to radicals. The radical-scavenging activity of flavonoids depends on their molecular structure and substitution patterns, particularly on their ability to stabilize the resulting phenoxyl radical via hydrogen bonding or resonance. Moreover, the C2-C3 double bond (structure shown in Table 2) conjugation with the 4-keto group, which is responsible for electron delocalization from the B-ring, enhances the radical scavenging ability [64]. The literature suggests that

one quercetin molecule can neutralize two free radicals (see Figure 10), which could explain its effectiveness in stabilization [31, 65]. Furthermore, quercetin's higher antioxidant activity can also be attributed to its lower molecular mass and bifunctionality in terms of active phenolic OH groups. When 0.25 wt.% AO is added to 1 g of mixture, quercetin (considering two active phenolic OH groups per molecule) provides 0.017 mmol of active phenolic OH groups per gram, compared to 0.0085 mmol for I1010 and 0.0058 mmol for tocopherol. Thus, the quercetin formulation contains approximately twice as many active phenolic OH groups as an equivalent concentration of I1010. This also explains quercetin's higher stabilization activity.

4.1 | Synergistic Antioxidant Interaction

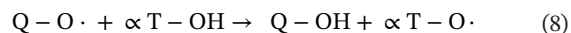
A combination of AOs can result in three distinct interactions: additive, synergistic, or antagonistic. Synergistic effects occur when combined AOs enhance oxidative stability more than the sum of their individual effects. Synergism can be influenced by multiple factors, including processing conditions, AO types, ratios, and concentrations.

Earlier studies on the synergistic interactions of AOs suggest that, in a combination of AOs with different antioxidation activities, the higher one can be regenerated by the lower one, and such combinations usually exhibit a degree of synergism [35, 66, 67]. The regenerative capability of AOs can be estimated by comparing their one-electron reduction potential (E°), which indicates the potential of an AO to donate or transfer a single electron [68]. It is possible that the combination of tocopherol and quercetin may exhibit synergistic effects through redox cycling. Quercetin has a lower redox potential (330 mV) than tocopherol (500 mV) [69]. Thus, tocopherol is likely to be regenerated by quercetin by donating one electron to tocopheroxyl radical (Equations 6 and 7). A similar mechanism is also explained by Zhang et al. [47, 70] and Pedrielli et al. [47, 70]



Another approach to estimate AO regeneration would be to compare bond dissociation enthalpies (BDEs), thermodynamic parameters that represent the energy required to break a bond. Comparing BDE values can indicate the likelihood of hydrogen transfer between AOs, analogous to E° , thereby providing insight into the possible regeneration mechanisms of the AOs. The BDE of the hydroxyl bond (OH) in tocopherol is 327 kJ/mol, whereas the weakest hydroxyl bond in quercetin possesses

345 kJ/mol [68, 71, 72]. This difference in BDE implies that it is more difficult for quercetin to donate hydrogen because it requires more energy to break the bond. Therefore, tocopherol protects quercetin against oxidation. This type of effect was observed by Becker et al. [50]. Thus, from a thermodynamic point of view, Equation (8) is more favorable [50].



Despite different mechanisms, either the repair and regeneration effect or the protective effect, the sample with quercetin and tocopherol demonstrated a clear synergistic effect, which makes it a suitable replacement for synthetic AOs.

5 | Conclusions

Mechanical recycling is an effective method of reprocessing PCPP. However, the inherent nature of mechanical recycling can inflict irreversible damage, known as thermomechanical degradation, which is detrimental to the polymer's thermal properties due to molecular chain scission and the generation of free radicals, often resulting in concomitant inferior mechanical properties. Such polymers are not suitable for upstream applications. AOs provide the most effective solution for preserving thermal properties by preventing or slowing down thermomechanical degradation. Both synthetic and natural AOs can be used to counter thermomechanical degradation. However, natural AOs provide a better, sustainable choice. Our study confirms that the natural AOs tocopherol and quercetin are more effective than synthetic AOs. Both can potentially replace synthetic AOs owing to their superior OIT and OOT properties. The DPPH assay and CI values also align well with the other experiments. This study further confirms that the combination of tocopherol and quercetin produces a synergistic effect, thereby elevating OIT and OOT characteristics. This combination also enables reprocessing of PCPP up to 5 times without compromising its thermal properties or melt strength.

This work contributes to the existing knowledge by providing clear experimental evidence of the superior performance and synergistic behavior of natural AO systems in recycled PP, highlighting their potential as sustainable replacements for synthetic AOs in circular polymer processing.

Author Contributions

Jeevan Tom Joseph: conceptualization, methodology, software, data curation, investigation, validation, visualization, writing – review and editing, writing – original draft. **Muhammad Irfan Qadeer:** conceptualization, methodology, investigation, validation, formal analysis, supervision, funding acquisition, visualization, resources, writing – review and editing. **Peter Leisner:** conceptualization, methodology, software, investigation, validation, supervision, project administration, resources, visualization, funding acquisition. **Mikael Skrifvars:** conceptualization, methodology, resources, supervision, project administration, investigation, validation, formal analysis, visualization, writing – review and editing.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

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