

# How to Measure Polymer Degradation? An Analysis of Authors' Choices When Calculating the Carbonyl Index

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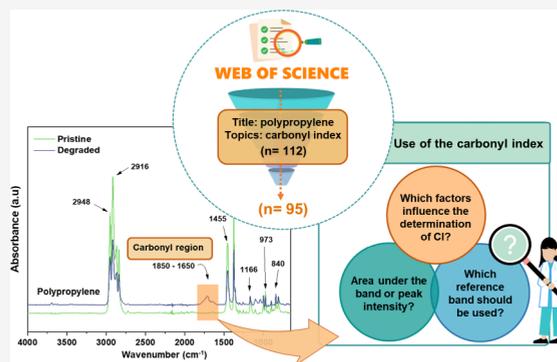
Supporting Information

**ABSTRACT:** The carbonyl index aims to measure the degradation level and is used in plastic degradation research as a proxy for the general degradation level of collected plastic pieces. According to the choices for carbonyl index calculation, comparison using this index is prevented and must be unveiled by the authors, which does not always happen. In order to study the proper usage of the carbonyl index, regarding the choice of the reference band and the usage of the band intensity or the absorption area, we systematically reviewed the methodologies used for polypropylene as a case study. Based on 95 studies gathered from 2000 to 2024, two main methods were used to determine the carbonyl index: the ratio between the carbonyl band area and the reference band area (33.68%) and the ratio between the highest intensity of the carbonyl band and the reference band (66.31%). The reference band of choice and the type of calculation method produce different carbonyl index values for the same spectra and mean different information, preventing comparison among works with different calculations.

**KEYWORDS:** polymer degradation, carbonyl index, polypropylene

## 1. INTRODUCTION

Anthropocene, defined by human-induced environmental transformations, presents a critical challenge: plastic pollution, one of the recent decades' most widespread and enduring features. As proof of its pervasiveness, plastic waste and its adverse effects have been documented in every iconic environment, from mountain tops to the bottom of the sea, across cities, and even suspended in the atmosphere.<sup>1</sup> Additionally, one of this era's main features is investigating and understanding the changes that occur on the surface of these materials.<sup>2</sup> The perception of such modifications is significantly relevant in the integrity of their applicability and the estimation of their harmful effects on the environment.<sup>2,3</sup> Once plastic materials are exposed to ecosystems, they are subject to adverse weather conditions, such as rain, solar radiation (ultraviolet), physical abrasion, and microbial effects. Such conditions can cause photodegradation, thermal degradation, wear, and abrasion on the surface of these materials and consequently cause embrittlement and fragmentation into smaller particles (micro and nanoplastics).<sup>3</sup> These processes cause chemical changes that degrade the surface of materials through a series of oxidative reactions in the polymer chain, modifying its composition, structure, and properties.<sup>4,5,44</sup> The chemical processes associated with polymer degradation are relevant for the studies related to the environmental impact of plastic pollution, like the study of fragmentation and formation of dissolved organics,<sup>50,54</sup> the development of methods to



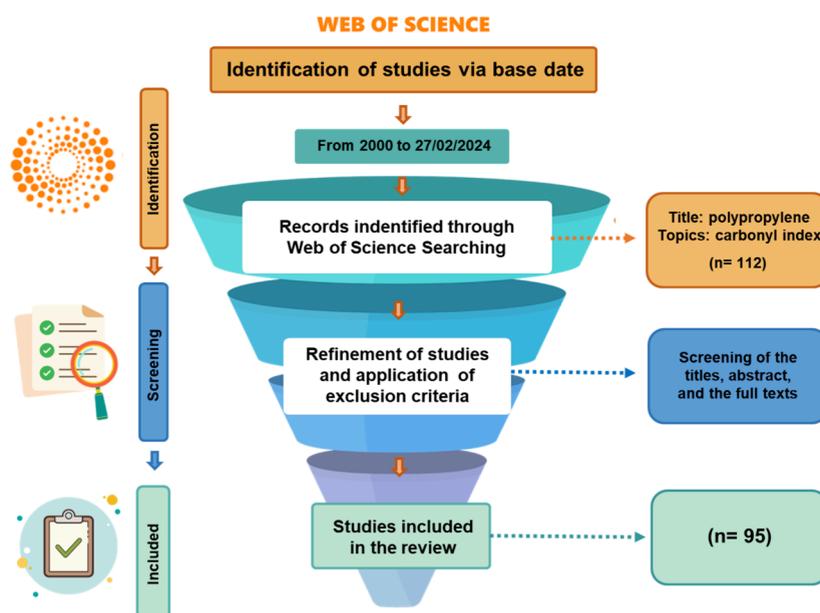
analyze micro and nanoplastics despite their degradation level,<sup>51</sup> and the influence of the weathering in the formation of biofilms on microplastics.<sup>52</sup>

One of the main techniques used to monitor chemical changes on the surface of plastic materials has been Fourier-transformed infrared spectroscopy (FTIR).<sup>6–8</sup> This approach allows the detection of functional groups of the substance through distinct absorption bands.<sup>6–8</sup> For this, it is customary to calculate the ratio between the area or intensity of a band that varies as a function of a stimulus to another not sensitive to the same stimulus, known as the reference band.<sup>6</sup> In plastics research, perhaps the most common example of this approach is the carbonyl index (hereafter called CI), which measures the intensity or the area of the carbonyl band compared with the intensity or the area of a reference band. From this perspective, the CI has been used for several purposes, such as the evaluation of resistance to degradation through extrusion processes,<sup>9</sup> the assessment of the resistance of recycled and reprocessed materials,<sup>10</sup> the development of stabilizers and formulations,<sup>11</sup> the prediction of the useful life of a material,<sup>12</sup>

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**Figure 1.** Pipeline for the literature gathering to find and analyze studies using the carbonyl index in polypropylene materials.

and the relationship between the level of degradation and the fragmentation potential.<sup>47</sup>

Initially suggested by Mellor et al.,<sup>13</sup> the CI specifically monitors the absorption band of carbonyl species as one of the main degradation products formed during the oxidative degradation processes of polymers. This region results from the convolution of at least three main bands,<sup>14</sup> attributed to photoproducts that absorb in the same region, that is, carboxylic acids in dimer form ( $\sim 1712\text{ cm}^{-1}$ ), esters ( $\sim 1735\text{ cm}^{-1}$ ) and  $\gamma$ -lactones ( $\sim 1780\text{ cm}^{-1}$ ). Using this approach, Rouillon et al.<sup>7</sup> reinforced the evidence that the formation of different carboxylic species and maximum absorption peak vary according to the degradation time. After 40 h of degradation, the formation of bands with maximum absorption peak began, referring to dimeric acids ( $1712\text{ cm}^{-1}$ ), which prevailed as a maximum until reaching 80 h of degradation. After 80 h, a progressive change in the maximum absorption of dimer acids to esters was observed, which prevailed until the full-time study (150 h). Although the  $\gamma$ -lactone species appeared within 40 h of degradation, at no point in the degradation experiment were these carbonyl species between the maximum absorption peaks. Furthermore, it was observed that the concentration of these degradation products is directly proportional to the degradation time when comparing similar formulations.<sup>7,14</sup>

Regarding the studies of plastic pollution from varied sources subjected to distinct weather exposures, it is impossible to estimate this exposition time, because there is no experimental control and no information about the type and concentration of stabilizers. In that case, CI can be a proxy of the degradation level but not of the degradation time. With the growing investigation on the impact of plastic materials in the environment and the related characterization, some studies still apply CI for this purpose.<sup>15–17</sup> For example, CI PP from microplastics found in sediments was used to relate the level of degradation to the season. In addition, the CI of PP microplastics found in marine sediments near the port of Cartagena (southeast Spain) was used to relate the level of degradation with the different marine environments where the sampling was carried out.<sup>15</sup> Celik et al.<sup>16</sup> determined the CI of

PP microplastic particles in several seas around Japan to correlate the level of degradation and the different regions. Additionally, some initiatives tried to correlate the CI determined by FTIR with the CI determined by Raman because of the advantages of the technique regarding easier sample preparation.<sup>53</sup>

To check the proper usage of the CI to monitor polymer degradation and whether the CI can be used for comparison among studies, we performed a systematic review of the literature to follow how it is calculated and used. To carry on the review, we chose polypropylene because of the two most produced polymers in the world,<sup>45</sup> polypropylene is the one more prone to photodegradation due to the tertiary carbon of each monomer in the main chain.<sup>46,47</sup>

## 2. METHODOLOGY

**2.1. Literature Search.** The literature search was performed on Web of Science with the word “polypropylene” in the title and the expression “carbonyl index” in the topics, which encompasses the title, the abstract, the author keywords, and the “keyword plus.” According to the Web of Science Help section,<sup>18</sup> “KeyWords Plus” indexes terms automatically generated from the titles of cited articles. The outputs were limited for the Language “English” from 2000 to 2024, February 27th. The search yielded 112 records assessed for eligibility by reading the titles and abstracts; we also read the text when necessary. The studies that did not calculate the carbonyl index were excluded. After the screening, 95 articles were gathered for analysis (Figure 1). The degradation type categorization was done by gathering equivalent expressions to the pertinent categories (details shown in Table S1).

## 3. RESULTS AND DISCUSSION

Since studies on plastic pollution usually cite or learn the calculation protocols from the polymer degradation research, we based our review on the general literature calculating the carbonyl index without limiting our search to words such as microplastic or pollution. Doing so, we gathered all the

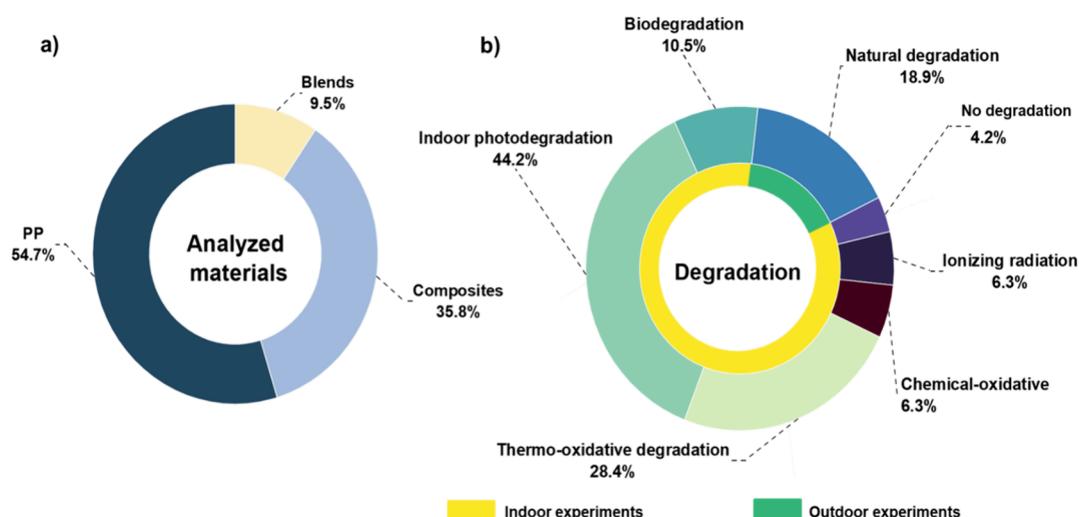


Figure 2. Percentage of publications distributed according to (a) material type and (b) degradation type.

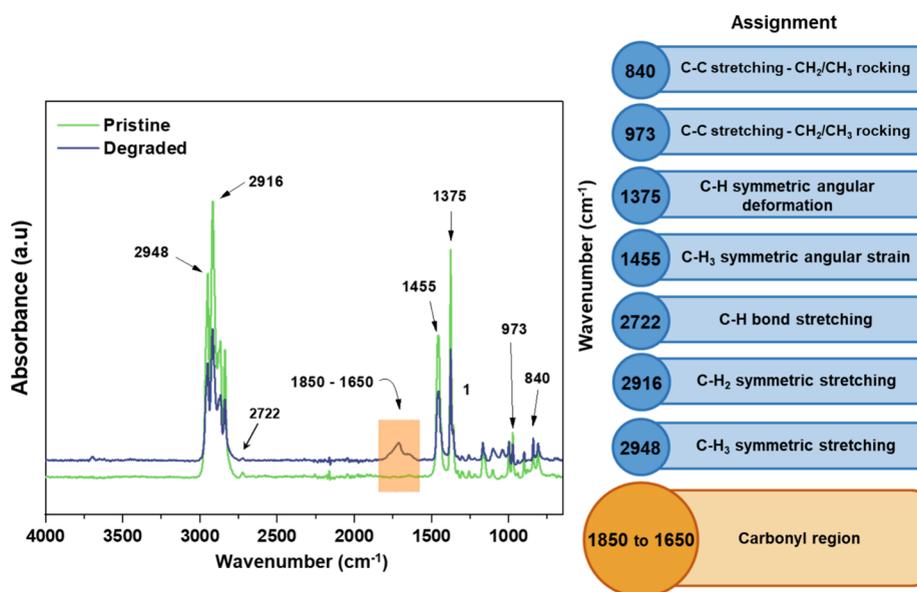


Figure 3. FTIR spectra of PP with the band's assignment.

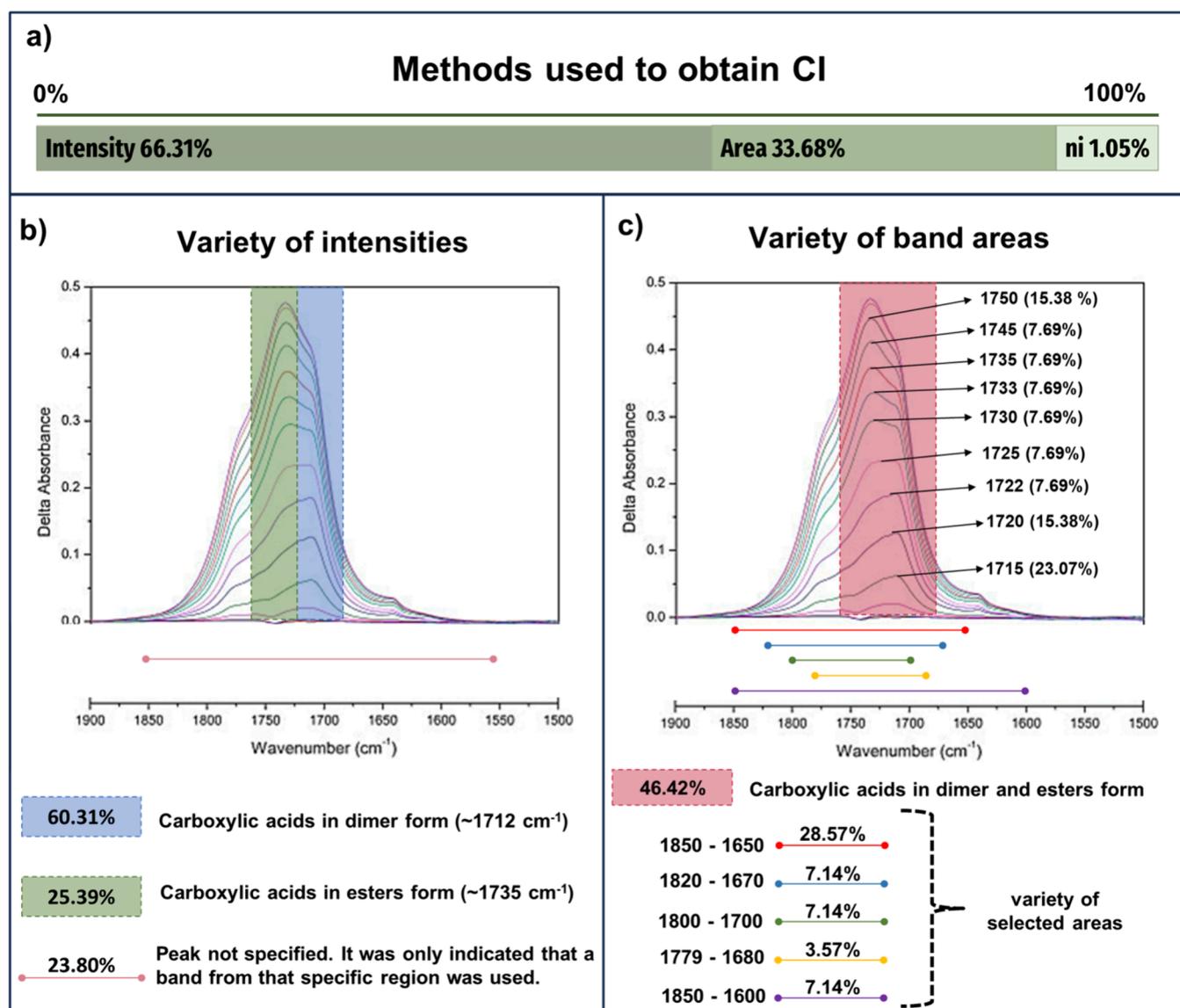
literature that calculated the CI and assemble the discussion about the choices of the authors.

**3.1. Framework Characterization of the Gathered Literature.** The distribution of the papers using CI to track the degradation level in polypropylene-based materials was 54.7% for polypropylene alone and 45.3% for multicomponent materials: 35.8% for composites and 9.5% for blends (Figure 2a). Figure 2b shows representative of the degradation types encompassing formation of carbonyl as part of the mechanistic evolution, with more than 40% of the studies in the context of indoor photodegradation and only 18.9% of the experiments performed in the outdoor environment. Notably, some studies performed more than one type of degradation, so the percentage will exceed 100% (Figure 2b).

The predominance of indoor degradation experiments (Figure 2b) is due to the need for better and more reliable assessment under accelerated and controlled conditions in a shorter period.<sup>11,12,19–42</sup> These experiments provide experimental data to indicate optimal parameters for the best adjustment of products and processes that delay degradation

and increase lifespan, improving recycling conditions and formulation.<sup>11,12,19–42</sup> On the other hand, exposition to the weather in outdoor degradation experiments allows better prediction of how properties change as a function of degradation time in realistic conditions, although the experiments take longer. While indoor experimental conditions provide essential information and predictions regarding the behavior of materials under such controlled experimental conditions, laboratory experimental tests do not encompass all conditions of a true-to-life environment. For instance, Rajakumar et al.<sup>43</sup> determined the CI of PP films that were directly exposed to the natural environment where they were exposed to the weather and the related conditions such as the entire solar spectrum radiation, not only the UV-A range, beyond rain, wind, and temperature variation as a function of the time, which influence the degradation.

According to the articles assessed, CI was the method employed to determine the degree of degradation of plastic materials. In these studies, the materials were exposed to mechanical forces, saline environments, solar radiation, rain,



**Figure 4.** (a) Distribution of studies that used the area or intensity. The value exceeds 100% because one of the studies used both methods, (b) variety of intensities used, and (c) variety of areas used. Reprinted with permission from ref 7. Copyright 2023 Polymer Degradation and Stability.

wind, and various temperatures. The environment where the material is exposed to degradation is a significant factor in evaluating CI, as the stressors to which plastics are subjected can directly influence the result. Photodegradation, for example, typically occurs on the surface of plastic materials, so the degree of deterioration can be higher on the surface of the plastic and lower in the inner regions of the material.<sup>7,16</sup> Then, a single material tends to have a deterioration gradient and, consequently, a CI gradient. Additionally, since photodegradation promotes the breaking of chemical bonds and the increase of crystallinity, the materials change, enabling cracks and peeling of the surface, which leads to fragmentation into smaller particles (micro and nanoplastics). This fragmentation can cause an underestimated CI value of the polymer matrix since the most degraded region undergoes fragmentation, leaving the matrix and maintaining the former inner layer, which is less degraded.<sup>16</sup>

**3.2. Determination of the Carbonyl Index.** Several sample features can interfere with the final spectra obtained by FTIR, like roughness, scattering radiation, or thickness, for

increasing the optical path. Since the entire spectrum will be homogeneously affected by those features, one approach to overcome this difficulty is the calculation of ratios between a band that changes as a function of a treatment one wants to measure and another band, called the “reference band,” which does not change as a function of this given treatment.

Figure 3 shows the characteristic spectrum of pristine and degraded PP with bands analyzed in the spectral range of 4000 to 500  $\text{cm}^{-1}$  and band assignments. In the pristine material, all the characteristic bands of PP are observed; however, bands are absent in the carbonyl region. On the other hand, in the degraded material, in addition to the characteristic bands, there is the convolution of carbonyl species bands that result from degradation products formed during the material degradation process. To calculate CI, the evolution of the absorption band of carbonyl species in the range of 1850–1650  $\text{cm}^{-1}$  is precisely monitored (highlighted in orange in Figure 3).

Figure 4a presents the results gathered from the 90 papers using CI as a measurement to monitor oxidative degradation. Here, 98.95% of the collected studies did inform the methods



The most used bands as reference were the bands at 974, 1456, and 2722  $\text{cm}^{-1}$  for the PP and those at 1456, 2722, and 2916  $\text{cm}^{-1}$  for the blends/composites.

CI obtained using different reference bands cannot be compared, since they use other chemical groups as denominators with varying coefficients of molar absorptivity. Given the mathematical operation, the values obtained can be greater or less in the CI calculation stage with an invariable numerator and variable denominator. Consequently, there will be a difference in CI values to underestimate or overestimate the degradation states of the materials. Considering this premise, the reference band used must not vary with the degradative processes to which the materials are subjected. However, it can be anticipated that the reference bands are not necessarily inert or insensitive to context change, as is the case of the  $\text{CH}_3$  band at  $\sim 1456 \text{ cm}^{-1}$ ,<sup>2</sup> which decreases in intensity due to the formation of volatiles containing the pendant  $\text{CH}_3$  groups, from PP. The degradation mechanism reported by Rouillon et al.<sup>7</sup> and Delprat et al.<sup>14</sup> indicate that the main volatile products are acetic acid and acetone, which carry the  $\text{CH}_3$  portion in which a large part of these products migrate from the polymer phase to the gas phase, causing the IR absorption of  $\text{CH}_3$  to decrease with exposure.

There is an increasing attempt to establish standards for CI use, application, and acceptable values. Consequently, the PAS 9017:2020 specification emerged to provide methods, deadlines, and criteria, demonstrating that polyolefins containing biodegradable additives can decompose in outdoor terrestrial environments without forming microplastics. To this end, PAS 9017:2020 uses CIs as one of the main parameters that determine whether this specific type of plastic decomposes and meets soil biodegradability requirements. According to the guideline, values greater than 1.0 in CIs after artificial or outdoor weathering indicate that the material will biodegrade in the soil without the formation of microplastic particles. However, although PAS 9017:2020 uses the second method identified in this study (area under the band) to monitor the carbonyl region, there needs to be a specification as to which reference band should be used. The lack of this information directly compromises the validity of this statement since, as previously discussed, the CI mathematical calculation is influenced by the reference band used. Furthermore, it was also discussed previously that several factors can influence the determination of CI, and many materials can exhibit a gradient of CIs. Therefore, it is necessary to consider several factors when standardizing the use of the CI.

Baseline correction is an important matter underestimated in the literature. From all 95 papers, 5 cited they performed a step of baseline correction, although with no details and only 1 work<sup>16</sup> described the details of the baseline. For a detailed discussion on the precautions and care about baseline correction and usage, we refer to the works of Zhang et al.,<sup>48</sup> Almond et al.,<sup>6</sup> and Yang et al.<sup>49</sup>

In summary, the method using the area for the CI calculation (33.68% of the choices) takes into consideration the carbonyl diversity, whereas the intensity method (66.31%) takes into consideration the predominant form of carbonyl at the time of the measurement, which might change as a function of time. Regarding the reference bands, the diversity is significant, but the predominance for area or intensity is the band at 1456  $\text{cm}^{-1}$ . Additionally, the choices for the reference band influence the CI value, preventing comparison among different works. Finally, due to the lack of standards, some

works use bands not assigned to the polypropylene structure as reference bands, which is inadequate and biases the results.

## ■ ASSOCIATED CONTENT

### Data Availability Statement

The data sets generated and/or analyzed during the current study are available in Table S3.

### Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acs.est.3c10855>.

Categorization by degradation type and by reference bands and list of all studies considered (PDF)

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### Author Contributions

R.S.G. and W.R.W.: Conceptualization, methodology, investigation, and writing—original draft. A.N.F.: Investigation, resources, writing—review and editing, supervision, project administration, and funding acquisition.

### Notes

The authors declare no competing financial interest.

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