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Some Thoughts on the Monitoring of Pristine and Aged Plastics by FT-IR Spectroscopy

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Abstract

Plastics can be identified by infrared-(IR)-spectroscopy. Often, the materials possess additives that confer special properties to them, such as elasticity, hardness, UV stability and color, but make it inherently more difficult to identify the polymer material associated. The inorganic salt calcium carbonate ($CaCO_3$) is one such additive that is used as filler, often in polythene-type plastics. Frequently, the IR absorption bands of $CaCO_3$ obscure the underlying bands of polythene. This may lead to misidentification of the material, especially in the case of microplastics (MPs), particles of less than 5 mm in size, where only small amounts of material are at hand. Over time, plastic material ages, where an automated identification of aged plastics can also lead to misidentification of the plastic, especially in the case of MPs. Here, the authors show that photo-oxidative aging does not only happen with polythene and polypropylene, but also with polystyrene and acrylonitrile-butadiene-styrene (ABS) co-polymer. Finally, the identification of the extent of photo-oxidation in the material can help monitor the integrity of plastics. Typical examples of monitoring the soundness of plastic chemical containers in a laboratory setting are given.

Keywords

Polythene, Polypropylene, Polystyrene, Acrylonitrile-Butadiene-Styrene, Microplastics, Oxidation, Infrared Spectroscopy, Calcium Carbonate, Plastic Traffic Cones, Aging Chemical Canisters

1. Introduction

Synthetic polymers are ubiquitous in our environment. Many of the objects we use daily are made from polymeric materials. Despite efforts to reduce the use of synthetic polymers, the production of many types of polymers continues to increase. Thus, the production of PVC (polyvinyl chloride) increased from 44.3 million metric tons in 2018 to a predicted 60 million tons in 2025. The market for polythene steadily grew from 92.8 million metric tons in 2015 to 116.4 million tons in 2024, with a slight decrease from 2019 to 2020 due to the shrinking economy during the COVID epidemic. Polypropylene, produced at 56 metric million tons in 2018 is predicted to reach 88 million tons by 2026, and finally the production of polystyrene is predicted to increase from 15.6 million metric tons in 2023 to 16.75 million tons in 2026. Altogether, the polymer production increased from 367 million metric tons in 2020 to a forecasted 404 million tons in 2025 [1]. The generated polymer waste increased from 352 million tons in 2020 to a forecasted 404 million tons in 2025. As of 2021, globally, only 9% - 10% of the polymer waste was recycled, 19% incinerated, while about 50% of the plastic waste ended up in landfills [2] [3]. This leaves 22% of polymer wastes that are discarded at non-controlled sites or in the environment [3].

Polymers that can be moulded into shape while soft and then set into a rigid or elastic form are called plastics. Oftentimes, in environmental sciences, the name "plastic" and "polymer" are used interchangeably, so that not all microplastics (MPs) meet the full scientific or engineering criteria for plastics. In the following plastic and polymer is used interchangeably, also, with the definition of macroplastics being polymeric particles of a size greater than 2.5 cm, with mesoplastics at 5 mm - 2.5 cm, microplastics (MPs) at 20 μm - 5mm, and nanoplastics (NPs) of less than 20 μm in size.

Usually, plastics contain a number of additives. These can include reinforcing fibers, fillers, processing aids, colorants/dyes, antimicrobial agents, photo stabilizers, antioxidants, flame retardants and plasticizers [4]-[7]. Inorganic additives to plastics are often used as fillers. They include silicates, glass fibers, talc, calcium carbonate (see below) as well as glass and ceramic microspheres [8].

In the open environment plastic degrades over time. This can occur through chemical processes such as photo-oxidation [9], enzymatic processes (biodegradation) [10] [11], and through physical processes such as through mechanical abrasion [12] or through temperature cycling [13] [14]. Many of these processes lead from the original macroplastic materials to meso-, micro-, and nanoparticular materials. While the complete degradation and/or mineralization [15] of plastics are slow [16], out of necessity the degradation proceeds through the production of MPs and NPs, which then degrade further. These MPs and NPs have become pervasive throughout all environmental compartments [17] [18]. They are ingested by different organisms [19], including livestock [20] and fish [21] and are known to have entered the human food chain [22] [23]. In fact, they have been found in many human organs including the uterus [24] and brain [25].

Over the last 30 years, efforts have been devoted to baseline assessments of MP contamination in the environment [26]-[28] and to the development of ideas for their mitigation and removal [29] [30]. Publications discussing the presence of microplastics in specific environmental compartments often provide a breakdown of the polymer types, with micro-Raman and micro-IR spectroscopy commonly used for identification. These studies typically present a selection of IR spectra. Interestingly, the spectra are almost always from pristine microplastics, meaning those that have not undergone degradation. This may be surprising and could be explained by the fact that authors select those spectra that best match known polymer types. It could also mean that oxidized microplastics are not readily identified, especially if the identification is automated. The identification of samples can also be hampered, when inorganic fillers obscure the spectrum of the polymer [31]. In the following, we will present such examples, where simple acid digestion of the samples is not sufficient to remove the inorganic filler material and subsequent IR spectroscopic analysis of such MP samples may well lead to the wrong identification of the samples as being of inorganic nature rather than microplastics. Also, examples of IR spectra of oxidized plastics made of polystyrene and acrylonitrile-butadiene-styrene are given that may make an automated identification difficult. Finally, the application of IR spectroscopy is demonstrated as a method for identifying oxidized plastic chemical containers that have failed or reached material failure.

2. Materials and Methods

Sampling

9 different materials were looked at for this study. 2 specimens (samples 1 and 2) were taken, one each from 2 different single-use plastic bags made of polythene that had been kept in a closed office at UAEU for exactly 78 months. At the point of sampling the plastic bags had degraded to such a degree that the samples can be classified as microplastic (MP). One sample each of aged acrylonitrile-butadiene-styrene (ABS) (sample 3) and of polystyrene (sample 4) were collected from the campus of the university (24.19562; 55.67835). These specimens can be classified as macroplastics. One specimen (sample 5) was collected from the Hudayriat Island beach, Abu Dhabi (24.40121; 54,37632). It can be classified as a polythene microfilm and most likely stems from a degraded plastic bag. One sample (sample 6) was taken from a degraded traffic cone, which was collected on the university campus (24.19513; 55.67805). This sample consisted of a large number of microparticles of polythene in a $CaCO_3$ matrix. Samples 7 and 8 come from broken plastic chemical containers, one holding glass wool (sample 7) and the other hydraulic anchoring cement (sample 8). Sample 9 is from an intact plastic chemical storage container for KOH pellets. Samples 7 - 9 were kept in a laboratory storage facility.

Infrared analysis

FT-IR spectra were recorded as KBr (Sigma Aldrich) pellets on a Perkin Elmer Spectrum Two spectrophotometer. The transmittance of the KBr sample pellet was measured in the range of 4000 to 500 cm^{-1} after 32 scans. When macroplastic samples were analyzed, some material from the surface of the sample was abraded with a 200 mm flat rasp (Magnusson®). The resulting plastic powders were measured as KBr pellets.

Acidic digestion

According to the FT-IR spectra, samples 1, samples 5, and samples 6 were found to have $CaCO_3$ content. These samples were stirred on a magnetic stirrer (Wisestir) in half conc. HCl [Honeywell, conc. HCl in distilled water (1:1, v/v), 3 mL for every 5 mg sample] for 2 weeks (without exchange of digestion medium, sample 1) and 3 days (with one exchange of digestion medium, sample 5), respectively, at rt. Afterwards, the samples were filtered through a glass fritte and washed with distilled water (3 × 5 mL for every 5 mg sample). The obtained material was collected and dried at 37˚C for 14 h (Ecocell MMM, Medcenter Einrichtungen GmbH). In the case of sample 6, $CaCO_3$ proved recalcitrant to dissolve. In this case, the sample was filtered off the acidic medium, washed with distilled water and dried as above. The dried material was crushed in an Agate bowl and submitted to a further round of digestion under the conditions stated above. The process was repeated 3 times. After the decalcified samples 1, samples 5, and samples 6 had been dried, they were subjected to FT-IR spectroscopy.

Assessing the oxidation index of polythene samples

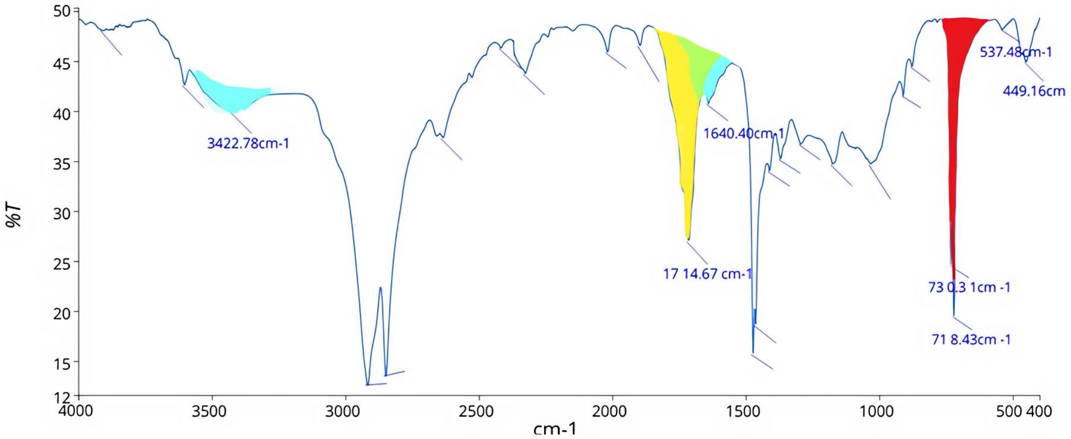

Figure 1. Typical FT-IR spectrum of an oxidized polythene material. In yellow, the band A associated with the oxidation of the polythene in form of C=O stretching vibrations, in red the band C associated with C-H rocking deformation vibration of polythene used as reference, in blue the band B due to H-O-H bending vibration of residual moisture in the material, in green the overlap of absorption bands A and B (according to ref. 32).

To quantify the oxidation of the polythene material, the area (A) under the absorption peak at approximately 1715 cm^{-1} (highlighted in yellow in Figure 1) was compared to the combined areas (B) under the absorptions resulting from the C-H rocking deformation vibrations centered around 718 and 730 cm^{-1} (highlighted in red in Figure 1).

Some samples showed traces of water, which caused an overlap between the ν_2 vibration mode (H-O-H bending motion) at 1595 cm^{-1} and the C=O stretching vibration at 1715 cm^{-1}. These overlapping bands were deconvoluted using Origin® software. The areas corresponding to both absorptions (highlighted in green in

Figure 1) were assigned 50% of the total area to the C=O stretching vibration, based on the weighing method used for quantifying the areas under the absorption bands, as described below.

The oxidation index (OxI) [32] was defined as OxI = A/B, with the areas under the absorption bands calculated using Origin® software. To verify the accuracy of the area measurements, the authors compared these calculations with results obtained by weighing cut-out areas from printed spectra using a micro-balance. Both methods showed comparable results. The OxI calculations presented in this paper were derived from the second method, with the weighing performed on a Kern ABT 220-5DM balance (readability: 0.1 mg/0.01 mg).

3. Results and Discussion

3.1. Identifying Polymers with $CaCO_3$ as Inorganic Additive

When taking FT-IR spectra of a wide range of products such as car-tire material [31] and single use plastic shopping bags, the signature spectrum of the additive calcium carbonate ($CaCO_3$) is obtained. Often, the intense bands of the carbonate ion obscure the IR bands of the polymer itself so that the polymer cannot be readily identified by IR spectroscopy. The major (strong) IR bands of $CaCO_3$ in form of calcite are found at 1430 cm^{-1}, 874 - 877 cm^{-1} and at 710 - 715 cm^{-1} [33]. Weak bands are found at 2510 - 2515 cm^{-1}, 1796 - 1799 cm^{-1}, 1020 - 1022 cm^{-1}. Polythene has its major bands at 1463 - 1474 cm^{-1} (CH_2 bending mode) and at 720 - 730 cm^{-1} (CH_2 rocking mode) [34]. The band of $CaCO_3$ centered at around 1430 cm^{-1} is very broad so that it can obscure the polythene band at 1463 - 1474 cm^{-1}.

$CaCO_3$ is commonly used in the plastic industry as a filler (often referred to as filler masterbatch or calcium carbonate filler) [35]. It typically appears as milky white granules, primarily composed of $CaCO_3$ and a resin base. It is favored for several key reasons:

Cost-effectiveness: $CaCO_3$ is abundant and readily available, making it a more affordable alternative to other filler materials, such as barium sulfate ($BaSO_4$). Sometimes, however, $BaSO_4$ is used as an additive in rubber materials. When used in plastics, calcium carbonate enhances the hardness, stiffness, dimensional stability, and overall processability of the final product.

Improved Processing and Stability: At the right concentration, calcium carbonate enhances the flow behavior of plastics, reducing the risk of warping and cracking during processing. Additionally, it improves the material's stability by increasing its resistance to UV light, preventing discoloration, and contributing to a longer lifespan. As a result, plastic products containing $CaCO_3$ maintain their appearance and functionality over longer periods of time.

In many studies on MP presence in the marine and terrestrial environment, MPs are separated by density separation, with organic material derived from organisms digested under oxidative conditions. In an oxidative digestion with H_2O_2/NaOCl, $CaCO_3$ is not affected. When MPs are isolated from tissue matter, such as from the gastrointestinal tract of fish, aq. KOH is often used in the digestion process

[36]-[38]. Again, $CaCO_3$ is not affected by digestion and will remain with the MP for examination. In a few instances, HNO_3 has been used in digestions to rid MPs of other organic constituents. Here, it could be expected that $CaCO_3$ reacts to $Ca(NO_3)_2$ (calcium nitrate) which is water-soluble [1.21 g/mL (20˚C); 2.71 g/mL (40˚C)]. $HClO_4$ (perchloric acid) has also been used [38]. Nevertheless, acids [39] are rarely used for digestion because of the worry of the destruction of certain types of MPs such as polyesters in the case of a complex matrix. Nevertheless, stirring the plastic/MP in aq. HCl is sometimes a solution, as can be seen in **Figures 2-3** and equation 1. In many cases, however, even simple stirring of the particles in half-conc. aq. HCl (hydrochloric acid) is not effective enough to remove $CaCO_3$ as water-soluble $CaCl_2$ (calcium chloride) after reacting according to equation 1 as much of the $CaCO_3$ is embedded in the hydrophobic polymer material or is indeed encapsulated by the polymer material and thus will not come into contact with aq. HCl to react. Even the digestion of films from single use plastic bags with half conc. HCl at rt does not always remove $CaCO_3$ completely as shown in **Figures 4-6**.

$$(1)\ CaCO_3 + 2\ HCl \rightarrow CaCl_2 + CO_2 + H_2O$$

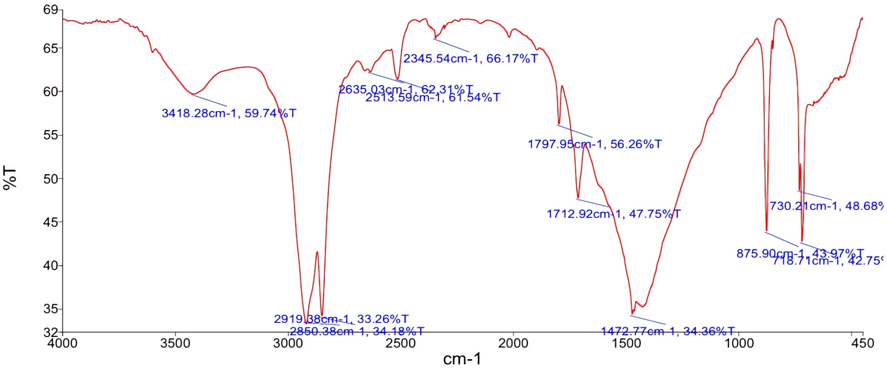

Figure 2. FT-IR spectrum of plastic microfilms stemming from a 78-month-old shopping bag (sample 1). The sample has had no pre-treatment. $CaCO_3$ is in evidence.

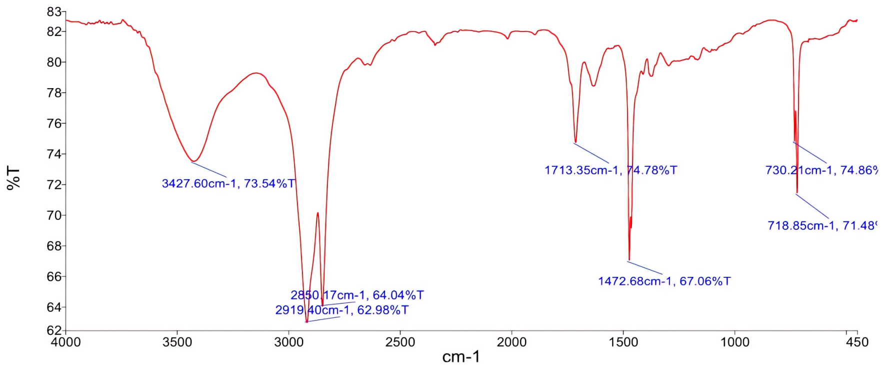

Figure 3. FT-IR spectrum of plastic microfilms stemming from the same shopping bag (sample 1) as the microfilms in **Figure 1**. The sample has been treated with half conc. HCl for 2 weeks at rt. The half conc. HCl as digestion fluid was not exchanged in the 2 weeks. The spectrum clearly shows oxidized polythene, with the $CaCO_3$ removed. The polythene material shows significant oxidation (OxI = 0.826).

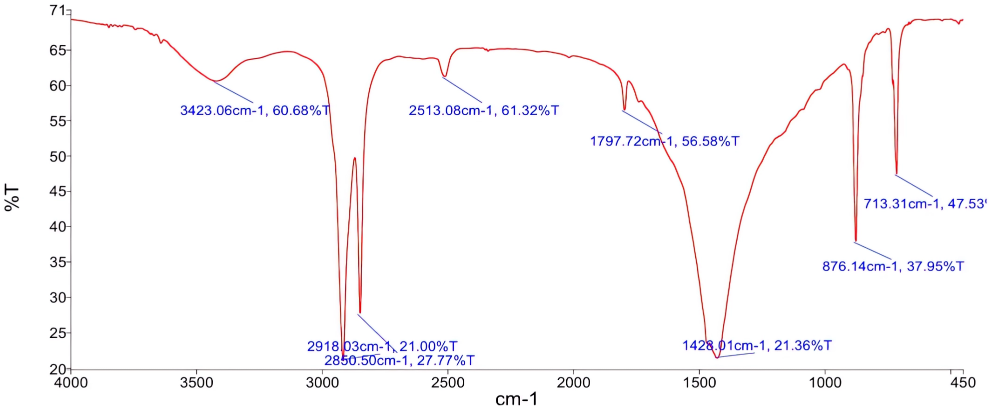

Figure 4. Microfilm (sample 5) found on Al Hudariyat Beach, most likely from a single use plastic bag. $CaCO_3$ is in evidence.

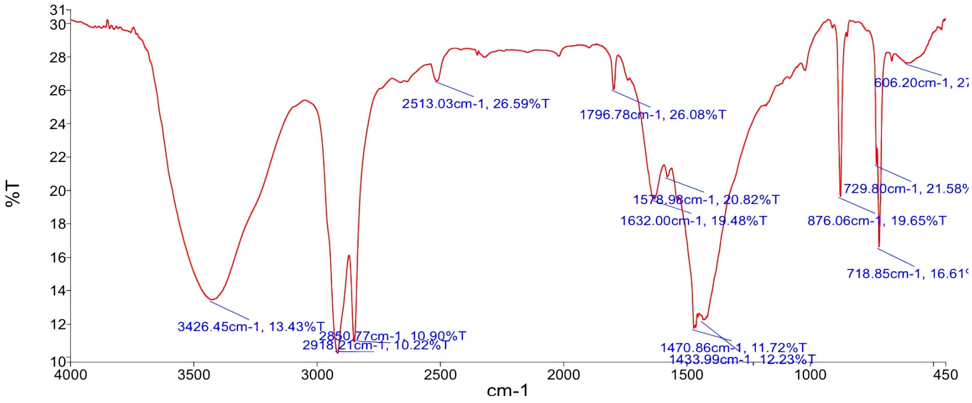

Figure 5. A microfilm from the same bunch of microfilms (sample 5) shown in **Figure 4**, stirred in half conc. HCl for 24h at rt. Although the microfilm was dried subsequently for 18h at rt, there is still moisture content, seen at 3426 cm^{-1} and 1632 cm^{-1}. The original absorption band centered at 1428 cm^{-1} has now visibly split into two bands with the one centered at 1470 cm^{-1} stemming from polythene.

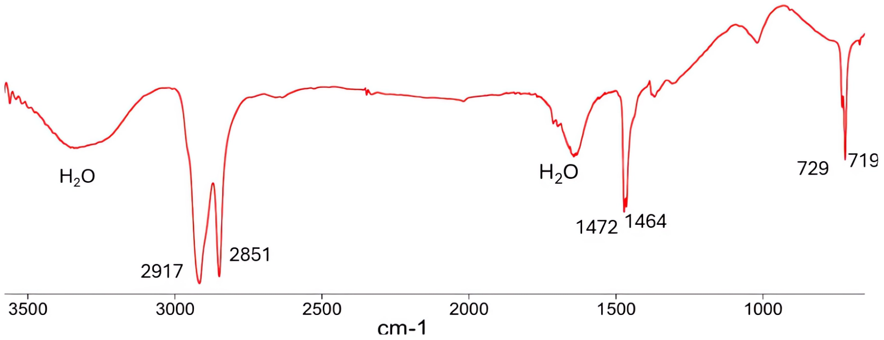

Figure 6. A microfilm from the same bunch of microfilms (sample 5) shown in **Figures 4-5** after a 3-day digestion in half conc. HCl. The $CaCO_3$ has been removed completely. The polythene microfilm shows little oxidation. (OxI = 0.174).

Another case in point is materials coming from a certain type of plastic traffic cones used in the Middle East, including in the United Arab Emirates (UAE). Plastic traffic cones can be made of various materials such as polyvinyl chloride

(PVC), rubber, or collapsible materials such as nylon, however, often simple cones are made of polythene with $CaCO_3$ as filler material [40] (**Figures 7-8**). Once exposed to the hot arid environment of the Middle East, volatile additives in the materials such as plasticizer and, in the case of tires, oils evaporate [41] leaving the polymer material with a relatively dry calcium carbonate matrix. At the same time, the polymeric material such as polythene in the case of the traffic cones undergoes photo-oxidation, which adds brittleness to the plastic material. Overall, at a certain age, traffic cones can break (**Figure 9**). It must also be noted that traffic cones are frequently not removed once they have fulfilled their intended purpose. Therefore, plastic cones made of polythene with $CaCO_3$ as additive are an important source of MP in the Middle East environment. Although MP contamination from polythene scaffolding and netting has been reported previously [42]—this source of contamination being very much in evidence in UAE, also—plastic traffic cones as a source of MPs (**Figure 10**) for the most part have been overlooked.

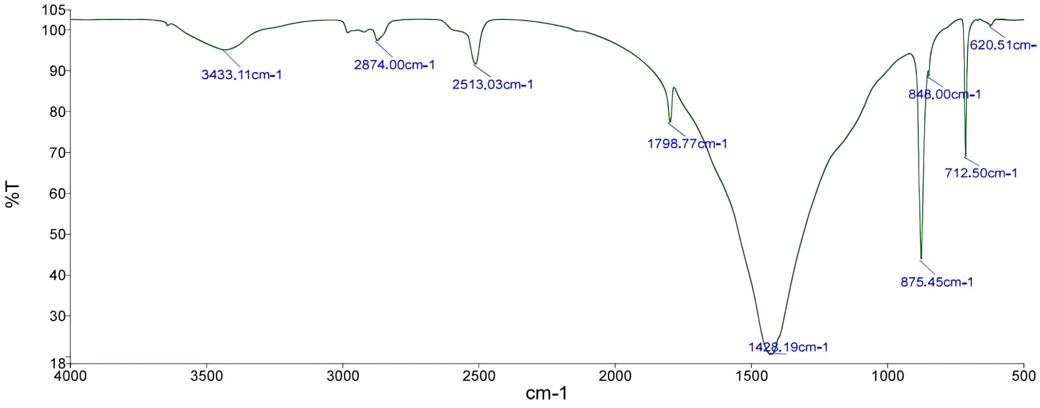

Figure 7. FT-IR spectrum of particles from polythene cone debris with embedded $CaCO_3$.

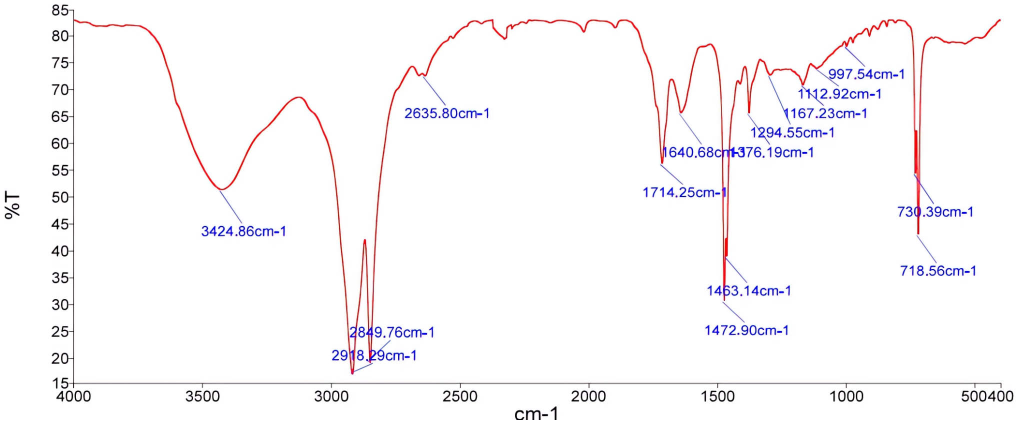

Figure 8. FT-IR spectrum of decalcified particles from the debris of a polythene cone after digestion in half conc. HCl (4 cycles). The spectrum shows heavily oxidized polythene (OxI = 0.912)

From **Figure 3** and **Figure 8**, it can be seen that the polythene is severely oxidized, even in the presence of $CaCO_3$. It must be noted that the polymeric material for the most part encapsulates $CaCO_3$ and is itself exposed to the open environment. Actual identification of the polythene material can involve 4 cycles of acidic digestion in order to remove the $CaCO_3$. The acidic digestion of the material does not affect the oxidation index.

Figure 9. Degraded polythene cones along streets in Al Ain, UAE. The degradation releases over 200 g polythene per cone into the environment. Most of the polythene will fragment further to microplastics.

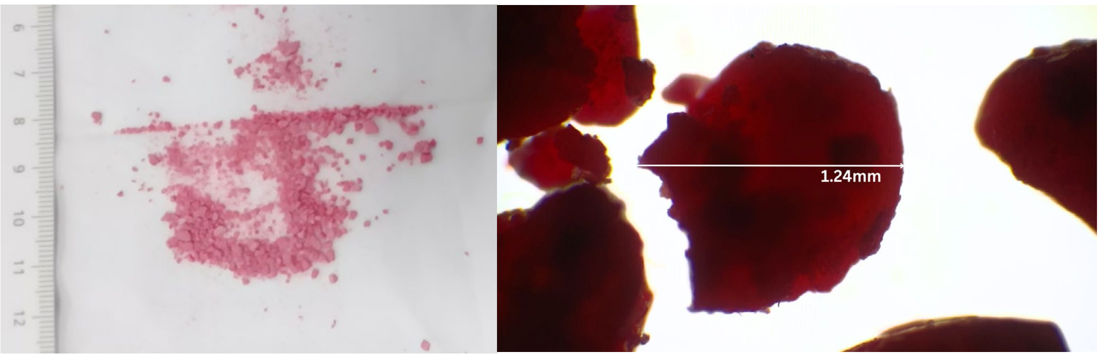

Figure 10. Microplastic particles collected from a degraded polythene cone. Photo with a Nokia C12 (left). Microphoto using a Leica ICC50 E microscope with a HI PLAN 4X0.10pol objective (right).

3.2. Identifying Photo-Oxidized Polymers

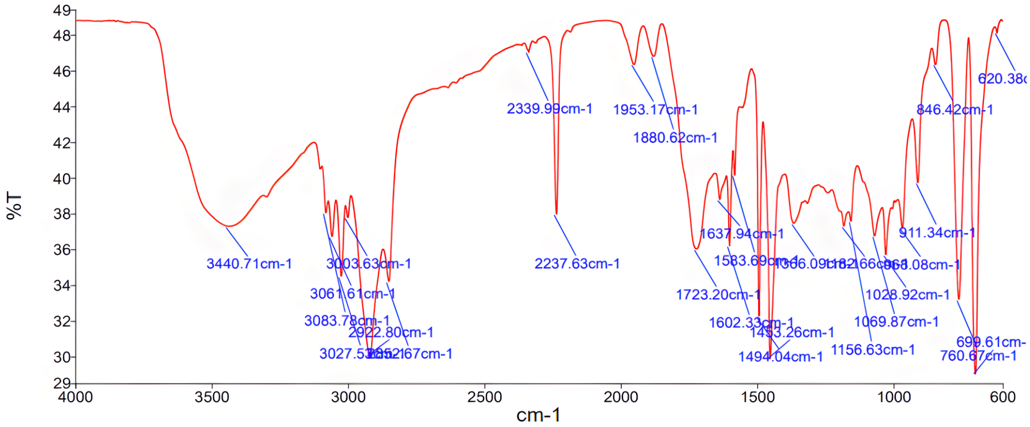

Figure 11. FT-IR spectrum of an oxidized ABS-based macroplastic

Degradation of plastic, specifically photo-oxidative degradation, alters the infrared spectrum of the material over time. It is realized from prior work that the rate of photo-oxidative degradation of plastics hinges on temperature, humidity, and the intensity and spectral distribution of the light reaching the plastic's surface, all of which depend on the location as well as the residence time of the plastic under investigation [32]. Most studies on these degradative changes of plastics have been carried out under controlled conditions in a laboratory environment [43]. These include [44] [45] near-IR spectroscopic measurements (14,000 - 4000 cm^{-1}; 0.7 - 2.5 μm wavelength) which are not the focus of this contribution. There are but few studies performed with plastics exposed to the environment [43] [46] [47] with all its variable conditions over time. From the authors' own studies [32], however, it could be ascertained that given a certain type of plastic material, the oxidative degradation of a number of particles of the same type set at the same location will give only a narrow distribution of oxidation indices, which then can be used to gauge the residence time of the individual plastic particles in the environment, once a calibration has been carried out. For the current contribution the oxidative degradation of acrylonitrile-butadiene-styrene (ABS) and of polystyrene (PS) were chosen. Thus, the FT-IR spectra of samples 3 and samples 4 show the oxidation of materials mainly consisting of ABS (**Figure 11**) and of PS (**Figure 12**). Both samples are macroplastics. Here, again with an automated matching of IR spectra to identify the polymer type, it is important to realize that polymers, incl. MPs, with long residence times in the environment may exhibit additional intensive bands that may complicate the identification. Therefore, it is beneficial to include the spectra of degraded polymer materials of known origin into a master database to match unknown samples against.

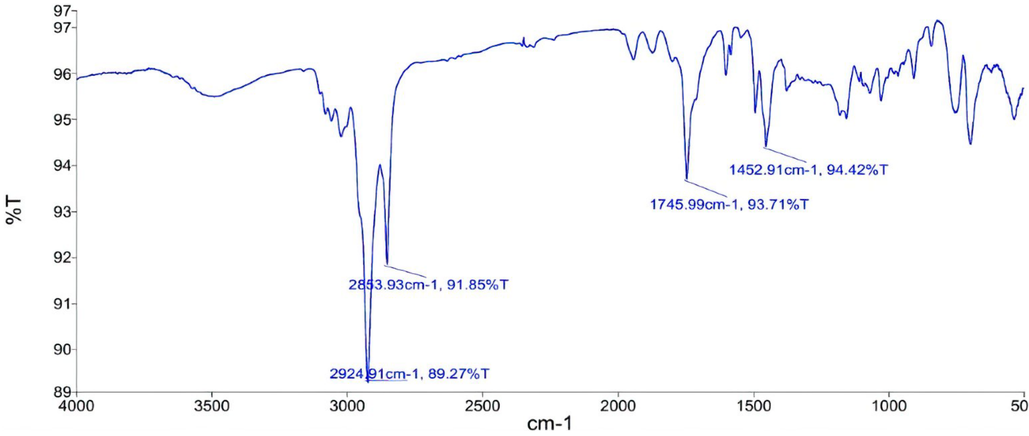

Figure 12. FT-IR spectrum of an aged, oxidized polystyrene mesoparticle. The C=O at 1746 cm^{-1} is unusually pronounced, so that material can be made of a co-polymer. The authors have found a number of other polystyrene particles where the band is less pronounced, but centered at or near 1746 cm^{-1}.

3.3. Use of the Oxidation Index (OxI) to Understand the Integrity of Plastic Containers for Chemicals in the Laboratory

Chemicals in a small laboratory setting are often stored in brown glass bottles when liquid or in polythene or polypropylene containers when solid. Even indoors, plastic containers oxidize, becoming brittle over time (**Figures 13-16**). This can lead to failure [48] of the container, leading to spillage of the chemical. It is usual that the plastic chemical container remains intact at least until the expiry date of the chemical it holds, however, there have been exceptions to this rule in our laboratory. FT-IR spectroscopy is a good way to check the integrity of the plastic material of the container. In our laboratory, the material of broken or damaged plastic containers have always shown an oxidation index > 0.4. **Figure 17** shows the IR spectrum of a 1-year-old polythene bottle carrying KOH pellets (sample 9). The oxidation of the material is minimal and the container is structurally intact.

Figure 13. 20-year-old container made of polypropylene holding glass wool (sample 7). The container has become brittle to the touch.

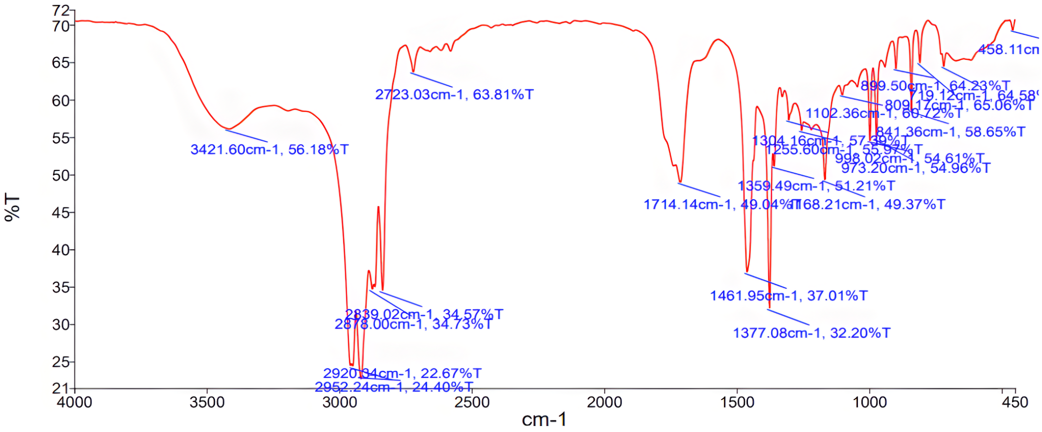

Figure 14. Polypropylene from the 20-year-old container shown in **Figure 13**. The polypropylene is heavily oxidized (OxI = 0.637).

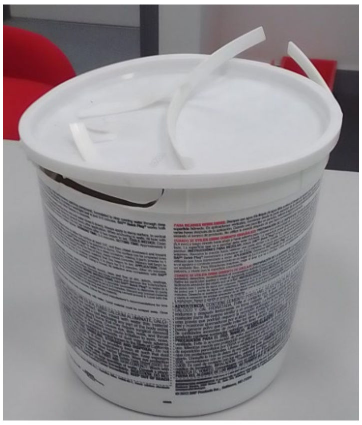

Figure 15. A 8-year-old container carrying hydraulic anchoring cement (sample 8). The polythene of the container has become brittle and the 10lb (4.5 kg) object can no longer by lifted by its plastic handle.

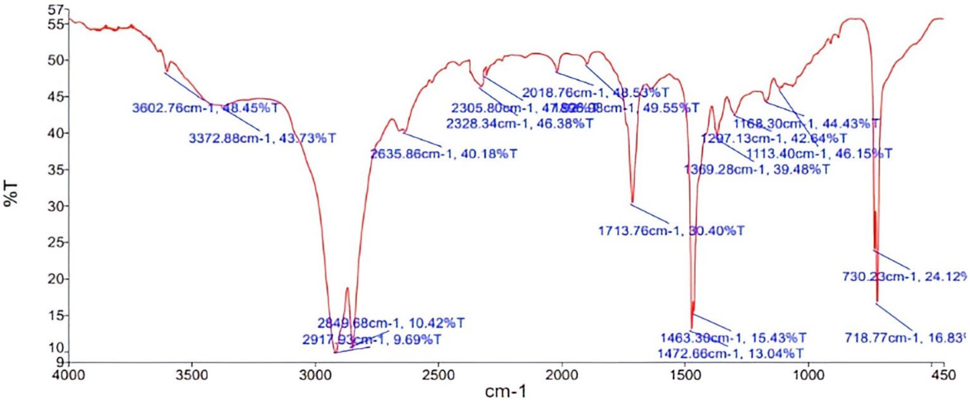

Figure 16. Polythene from the 8-year-old container is shown in **Figure 15**. The polythene is heavily oxidized (OxI = 0.448)

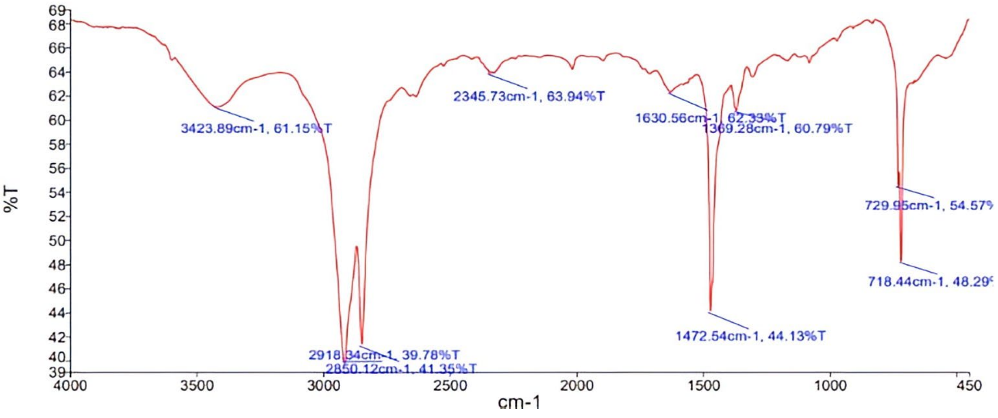

Figure 17. In comparison to the figure above, this is a FT-IR spectrum of a 1-year-old potassium hydroxide (solid KOH pellets) container made of polythene (sample 9). Little oxidative degradation can be noted (OxI = 0.078).

4. Conclusions

Additives in plastics and aging of plastics can complicate the identification of the plastic type of a sample. The paper showed one example of each, $CaCO_3$ as additive and the photo-oxidative degradation of polythene, polypropylene, polystyrene and acrylonitrile-butadiene-styrene. In the case of analyzing $CaCO_3$ containing plastics, including MPs, $CaCO_3$ content is not reduced by oxidative or basic digestion methods. Even digestions under acidic conditions necessitate long reaction times to completely eliminate $CaCO_3$ content. $CaCO_3$ content in a microplastic can lead to its wrong identification as a non-polymer particle, if FT-IR spectroscopy is used. Typical items that lead to large numbers of $CaCO_3$ containing particles are polythene traffic cones, which this paper identifies for the first time as a source of microplastics in the environment.

Over time, polymers such as polythene and propylene, but also polystyrene and acrylonitrile-butadiene-styrene co-polymers oxidatively degrade which makes them more prone to fragment. Such oxidized polymers may be less straightforward to analyze, if an automated system is used to match the obtained spectrum with a spectra database. Therefore, it is recommended that spectra databases include spectra of polymers with different degrees of (oxidative) degradation as standards to match unknowns against.

While aging of plastic may make the determination of the plastic type by FT-IR-spectroscopy more difficult, FT-IR spectroscopy may be helpful in determining the age and integrity of plastic material, when the plastic type is known. Chemicals are often packaged in plastic containers. These plastic containers frequently made of polythene or polypropylene have a certain lifetime and degrade oxidatively. Agreeably, chemicals have an expiry date, and should be exposed of at that time. Nevertheless, plastic containers have been found to fail before the expiry of the chemical. Here, the integrity of the polythene/polypropylene containers can be monitored by FT-IR spectroscopy. Damaged or cracked containers invariably have been found to have an appreciable oxidation index of > 0.4.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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