

1 Investigating the Potential Release of Microplastics from Recycled Plastic  
2 Modified Asphalt Pavement

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12 **Abstract**

13 Due to the ever-increasing volume of plastic waste, researchers are exploring alternative end  
14 destinations for recycling and repurposing, with civil infrastructure systems seen as promising  
15 hosts. One area that has seen exponential growth in research interest is Recycled Plastics Modified  
16 Asphalt (RPMA) pavement. Despite significant progress in this field, several issues remain  
17 relatively underexplored, including the potential release of microplastics from RPMA under stress  
18 conditions. This study attempts to help bridge this gap. RPMA and control asphalt pavement  
19 samples were prepared in the laboratory and mechanically tested using standard performance tests,  
20 including the Hamburg Wheel Tracking Test (HWTT) and Moisture-Induced Sensitivity Test  
21 (MiST), to simulate long-term aggravated service conditions. RPMA used an agricultural Low-  
22 density Polyethylene (LDPE) as the modifier. Leachate solutions with debris from these tests,  
23 along with background solutions and samples of raw materials, were screened for microplastic  
24 presence using Laser Direct Infrared Imaging (LDIR) and Attenuated Total Reflectance (ATR).  
25 The analysis revealed a variety of polymeric and non-polymeric particles across all tested samples.  
26 However, the quality of identification and the percentage of polymeric particles generally remained  
27 low. Pervasive polymeric background contamination, coupled with challenges in spectral  
28 similarities between different ingredients, complicates efforts to definitively determine whether  
29 RPMA specifically released microplastics, as various types of polymeric particles were detected  
30 and identified in the control samples as well. Future studies are recommended to implement more  
31 rigorous contamination controls and to incorporate complementary analytical techniques to  
32 improve identification confidence.

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36 Key Words: Microplastics, Recycled plastics modified asphalt (RPMA), Leachate, Laser Direct  
37 Infrared Imaging (LDIR), Attenuated Total Reflectance (ATR), Hamburg Wheel Tracking Test  
38 (HWTT), Moisture-Induced Sensitivity Test (MiST)

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## 1 Introduction

2 Plastics are widely used because of their favorable properties such as durability, strength,  
3 lightweight, and cost-effectiveness (1, 2). However, with a global plastic recycling rate of less than  
4 10% (3), their widespread use has led to significant environmental challenges. Hence, waste  
5 plastics are actively becoming a global concern (4–10). Recycling efforts aim to mitigate this issue  
6 by identifying sustainable end destinations for waste plastics, including their application in asphalt  
7 pavement (1, 9, 11–14). This approach is aligned with the use of other waste-based materials in  
8 asphalt pavement such as Reclaimed Asphalt Pavement (RAP) (15, 16), Reclaimed Asphalt  
9 Shingles (RAS) (17), and Crumb Rubber (CR) (18–20). Despite recent progress, the use of  
10 recycled plastics in asphalt pavement still faces several unresolved challenges (9). These include  
11 but are not limited to intermediate and low-temperature performance issues (1), storage stability  
12 (21, 22), workability (23), repurposing and recycling of recycled modified asphalt, effect on the  
13 asphalt plant operation (24, 25), potential emission of harmful gases during preparation (4, 10, 26),  
14 and potential release of microplastics during service (27). While some of these issues are receiving  
15 global attention and are being extensively researched, the potential release of microplastics from  
16 Recycled Plastics Modified Asphalt (RPMA) remains comparatively underexplored. However, as  
17 roads are constantly exposed to weathering and mechanical degradation that generate roadside  
18 dust, concerns about the potential release of microplastics from RPMAs are increasing (28).  
19 Although microplastics have long been found to accumulate in soil and bodies of water (29),  
20 research on the release of microplastics from RPMAs is limited. Likewise, there are no established  
21 standards or best practices for evaluating microplastic release from RPMAs. Researchers in this  
22 field are exploring various methods to assess this risk potential. Based on the limited literature, the  
23 prevailing assumption is that plastic-modified asphalt pavements could shed microplastics  
24 (fragments of plastics smaller than 5 mm in size) during their service life and these particles could  
25 end up in the aquatic environment by rainfall and surface runoff. Existing studies primarily focus  
26 on 1) whether RPMAs release microplastics, and 2) how significant this source is compared to the  
27 other road-related sources of microplastics such as particles from vehicle tire wear and polymeric  
28 paint used for road markings (27, 30). Typically, road microplastic studies involve collecting  
29 random samples of roadside dust and debris from various locations along the study area which are  
30 then analyzed for microplastic content using a range of techniques (30–34). However, this  
31 approach is less applicable for roads modified with recycled plastics, as such roads are not yet  
32 widely implemented. As a result, alternative laboratory-based methods are being developed to  
33 assess the potential release of microplastics from RPMAs.

34 One approach involves the use of Hamburg Wheel Tracking Test (HWTT) samples, where the  
35 solution and debris from both control and plastic-modified asphalt samples are collected after the  
36 test for quantitative and qualitative analysis of microplastic presence. Duan et al. (27) investigated  
37 the potential for the production of microplastic from plastic-modified asphalt pavement using  
38 linear low-density polyethylene (LLDPE) as an asphalt modifier. Within the conditions of their  
39 test, their results indicate that plastic-modified asphalt pavement could produce microplastics,  
40 however, about three orders of magnitude less than microplastics released from vehicular tire wear  
41 for a comparable amount of asphalt pavement.

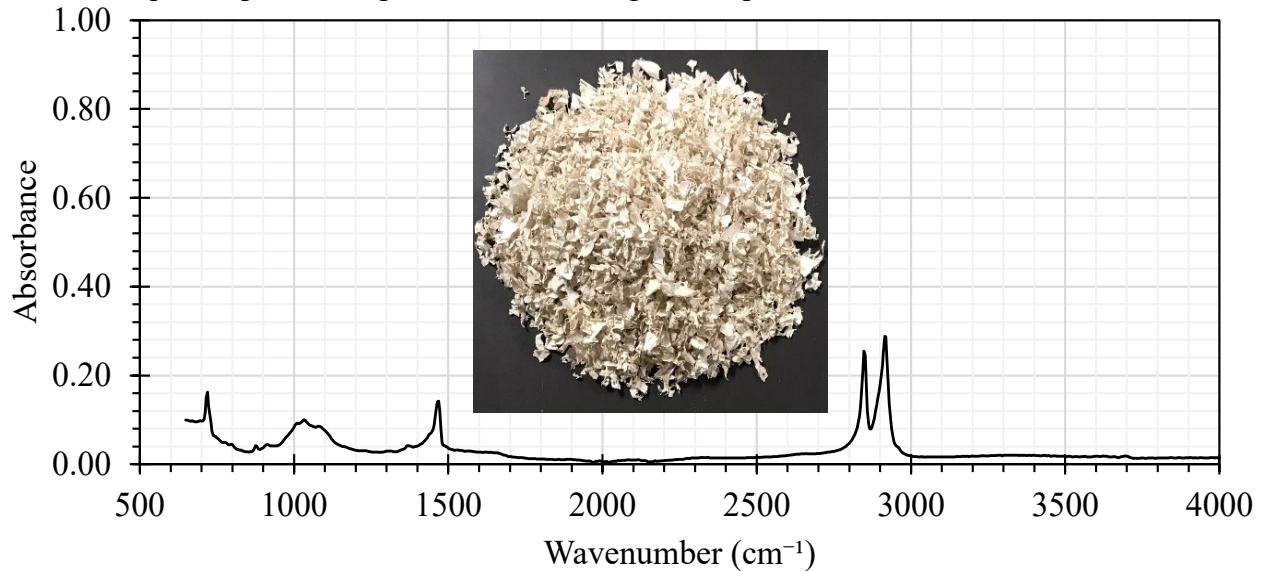
42 Similarly, several studies have confirmed the presence of significant amounts of microplastics in  
43 roadside dust from conventional roads (i.e., roads not modified with plastics) across various  
44 countries (31–34). They are primarily associated with wear from vehicle tires, road marking paint,  
45 and other plastic materials used on the roads. Therefore, the objective of this research is to

1 investigate the potential release of microplastics from Recycled Plastic Modified Asphalt  
2 Pavements under aggravated service conditions simulated in the lab.

### 3 **2. Material and Methodology**

#### 4 **2.1 Materials**

5 PG64-22 with a specific gravity of 1.028 was used as the base binder to prepare asphalt pavement  
6 specimens. Fine and coarse aggregates were sourced from a limestone quarry in central  
7 Pennsylvania. Specific gravities and absorption properties of the aggregates were 2.649, 2.655,  
8 1.19%, and 0.60% for fine and coarse, respectively. The RAP used throughout this project was  
9 also sourced from projects in central Pennsylvania. An anti-stripping agent of the commercial type  
10 KoHere AS-700 was used for all the mixtures. Recycled plastics used in this study were shredded  
11 Low-Density Polyethylene (LDPE) from agricultural films. Besides the supplier confirmation, the  
12 LDPE type was also verified using infrared (IR) spectroscopy that matched it to a polyethylene  
13 (chlorinated) wavenumber fingerprint with a match quality of 87.309% (Major spectral peaks:  
14 dominant C-H stretching regions between 2800  $\text{cm}^{-1}$  and 3000  $\text{cm}^{-1}$ ,  $\text{CH}_2$  bending at around 1400  
15  $\text{cm}^{-1}$  to 1500  $\text{cm}^{-1}$  and  $\text{CH}_2$  rocking at around 700  $\text{cm}^{-1}$ ). The IR results with an image of plastic  
16 particles are shown in Figure 1. The size of the shredded flakes ranges from plastic dust of a few  
17 microns to plastic pieces of up to 1.00 cm of irregular shapes and nonuniform sizes.



18  
19 Figure 1: IR spectra and an image of the type of LDPE used for this study

#### 20 **2.2 Methodology**

21 To study the potential release of microplastics from recycled plastic-modified asphalt pavement  
22 under service conditions, solutions with debris and sediments (leachate) were collected from  
23 conventional asphalt pavement mechanical tests such as the Hamburg Wheel Tracking Test  
24 (HWTT), and Moisture-Induced Sensitivity Tester (MiST). While the HWTT method is used by  
25 at least one other researcher (27), it is likely that MiST is not used for this purpose as of yet and  
26 hence introduced as novel procedure in this study. The collected leachate was then evaluated for  
27 microplastic presence using microplastic characterization techniques such as Laser Direct Infrared  
28 Imaging (LDIR) (Agilent 8700 LDIR Chemical Imaging System with Microplastics Starter 1.0

1 and Clarity v1.5.58 software) and Attenuated Total Reflectance (ATR) (Cary630 with MicroLab  
 2 v5.7 software). The overall research methodology is presented in Figure 2.

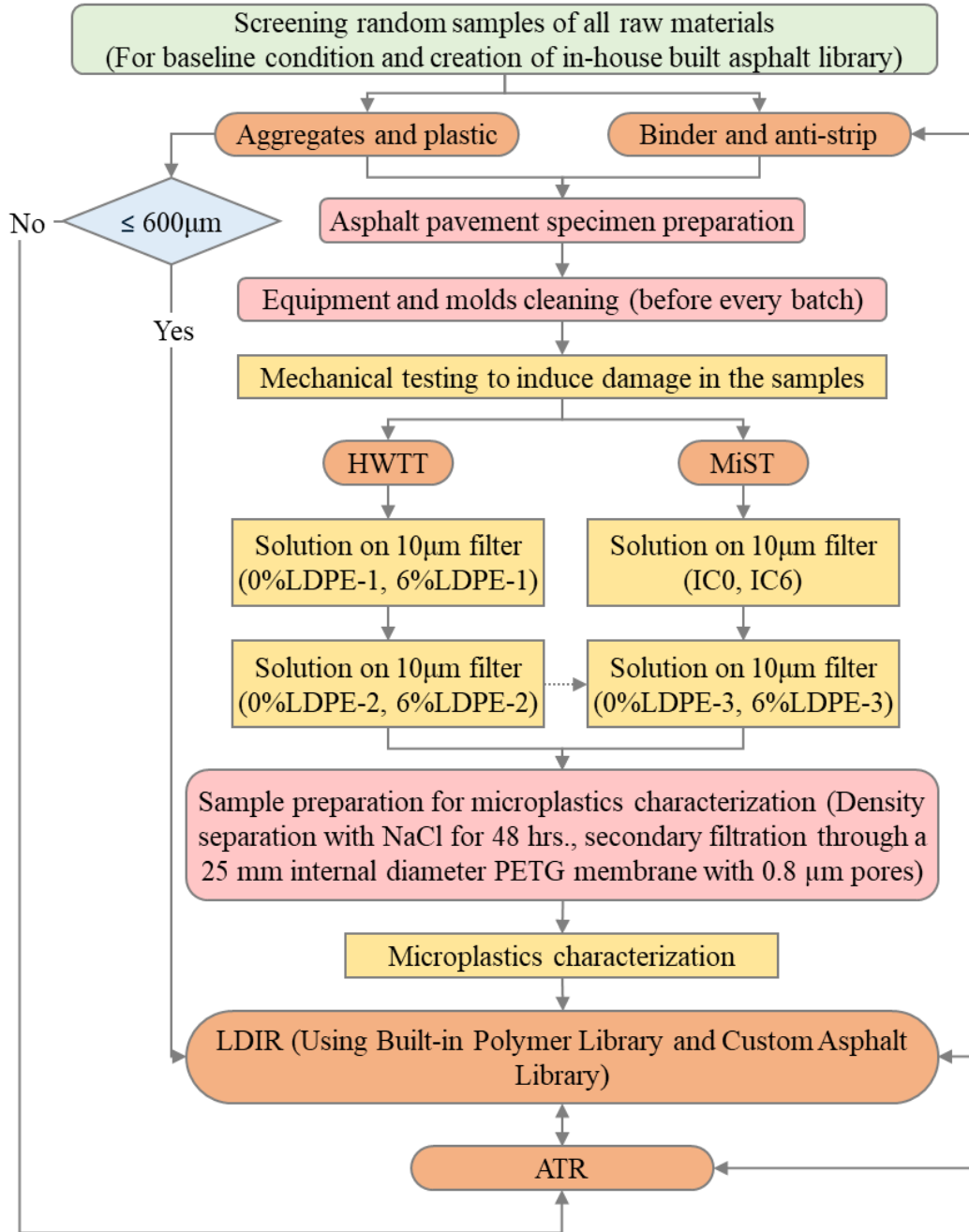


Figure 2: Research methodology flowchart

5 LDIR analysis was performed using two reference libraries: the built-in polymer library provided  
 6 by the LDIR instrument and a custom asphalt library developed specifically for this study. All  
 7 microplastic characterizations were carried out at the Institute of Energy and the Environment at  
 8 Penn State (PSU-IEE). Spectra for the custom asphalt library were collected using ATR analysis.  
 9 For each asphalt ingredient (raw material), an average of seven spectra were collected and  
 10 imported into the LDIR system under the ingredient's name, rather than its chemical composition,  
 11 as in the built-in polymer library. For example, while the built-in library might identify a 0.075

1 mm fine aggregate particle as silica, the custom library will identify it as “Sieve #200,” reflecting its role in the asphalt mix rather than its chemical identity. As a result, particles matched using the built-in library are reported by their chemical composition, whereas those matched using the custom asphalt library are identified by ingredient name.

### 5 **2.2.1 Raw Materials Screening**

6 Before the preparation of asphalt pavement specimens for HWTT and MiST tests, random samples  
7 from the bins of the raw materials were screened for microplastic presence for the creation of  
8 baseline condition and the custom asphalt library. This included samples of PG64-22 binder, anti-  
9 stripping agent (ASA), LDPE particles, and aggregates (9.5mm, 4.75mm, 2.36mm, 1.18mm,  
10 0.6mm, 0.3mm, 0.15mm, 0.075mm and < 0.075mm). PG64-22 and ASA were screened using both  
11 LDIR and ATR. On the other hand, while all the aggregates were screened using the ATR, some  
12 were not suitable for LDIR testing due to their sizes. Hence, a cutoff was set at Sieve #30 (600  
13  $\mu\text{m}$ ) or smaller for LDIR tests. Establishing baseline conditions was essential to determine whether  
14 any traces of plastic, particularly polyethylene-based materials, were present in the raw materials  
15 before the addition of LDPE as a modifier.

16 For the LDIR analysis of the aggregates, approximately 1mg of each material from the random  
17 samples was transferred to a 2000  $\mu\text{L}$  amber glass vial using a micro spatula which was followed  
18 by the addition of 1000  $\mu\text{L}$  of 91% ethanol into the glass vial by a micropipette. Subsequently,  
19 about 50  $\mu\text{L}$  of the solution was transferred to Low-E glass slides and was allowed to dry at room  
20 temperature for 5 minutes, before it was used for LDIR analysis. On the other hand, for the LDIR  
21 analysis of binder and anti-stripping agent, droplets of each material were directly deposited on  
22 Low-E glass slides used for the LDIR analysis. The sample with the binder droplets was allowed  
23 to cool down before the analysis. In both cases, the sample-containing slides were then attached to  
24 a mobile stage and then inserted into the LDIR instrument sample bay. The microplastics  
25 characterization processing method was then applied to the selected region of the slides where  
26 materials were deposited. The LDIR instrument detected every particle in the selected region and  
27 measured their physical attributes such as width, height, diameter, aspect ratio, area, perimeter,  
28 eccentricity, circularity, and solidity. Subsequently, it created a spectrum of each particle and  
29 identified them by comparing their spectrum to a selected library of a wide range of polymeric and  
30 non-polymeric materials and selecting the best match (based on the highest match quality between  
31 0% to 100%).

32 Likewise, for the ATR analysis of the aggregates and the LDPE, individual particles of each  
33 sample were positioned in the center of the ATR machine stage under the diamond crystal.  
34 Subsequently, the ATR anvil was lowered to ensure adequate contact between the sample and the  
35 diamond during spectrum analysis. For the binder and anti-stripping agent, a droplet of the material  
36 was deposited directly on the ATR diamond. To make sure that the intensity of the spectra was not  
37 reduced, the entirety of the diamond was covered with the sample liquid. Spectra were collected  
38 once adequate distribution of the sample was ensured. In all cases, once the spectra were collected  
39 it was then compared against the ATR search library that contains the most common thermoplastic  
40 polymers to find a match or several matches with a match quality like LDIR. The ATR machine  
41 stage, the anvil and diamond were cleaned with 89-91% ethanol using a cotton-tipped applicator  
42 before and after each test. Tests with binder samples required an extra step of cleaning with toluene  
43 or a citrus-based solution to completely remove any binder residue from the diamond and  
44 recleaning with ethanol solution. It should also be noted that although the aggregates were screened

1 for microplastics separately based on their sieve sizes, they come from the same limestone quarry.  
2 They were sieved in the lab for the preparation of asphalt pavement samples.

### 3 **2.2.2 Asphalt Samples Preparation**

4 The mix design presented in literature (23) with adjustment of 5.9% optimum binder content,  
5 0.25% anti-stripping agent and nominal maximum aggregate size was used to prepare asphalt  
6 pavement samples for this research. Before the mixing process, the aggregates and the binder were  
7 preheated at 160 °C for 2 hours, while the RAP materials were preheated at 110 °C for 1 hour.  
8 Upon completion of the preheating process, the aggregates and the RAP were blended for 90  
9 seconds using an asphalt mixer drum, followed by the addition of PG64-22 and mixing for another  
10 90 seconds. RPMA specimens were prepared using the dry mixing method where 6% shredded  
11 LDPE particles were added directly to the mix during the second 90 seconds of mixing time. After  
12 mixing, the asphalt mixtures were short-term aged at 135 °C for 2 hours following AASHTO R30  
13 standards and subsequently compacted using a Superpave Gyratory Compactor (SGC) into  
14 cylindrical disks of 60 mm thick and 150 mm in diameter, intended for HWT and MiST tests.

### 15 **2.2.3 Hamburg Wheel Tracking Trest**

16 The Hamburg Wheel Tracking Test (HWTT) was conducted following AASHTO T324. However,  
17 the objective of the HWTT in this study was not to evaluate the rutting resistance or moisture  
18 sensitivity of the specimens but rather to create road-like extreme damage to the specimens to  
19 induce microplastic leaching, if any. The HWTT is designed to subject the test samples to an  
20 aggravated aging and loading condition that simulates the wear and tear experienced by asphalt  
21 pavements over several years in the field but within hours in the lab. This is achieved by repetitive  
22 loading of steel wheels, moving back and forth for up to 20,000 passes on the test samples, which  
23 typically results in measurable and visible damage, often releasing debris and sediments. All the  
24 HWT tests were conducted in the water bath to maintain the testing temperature at 50 °C. The  
25 HWTT equipment with its molds and fixtures was washed and cleaned thoroughly before every  
26 test with a commercially available detergent for stainless steel applications. An image of the  
27 cleaning process is presented in Figure 3.

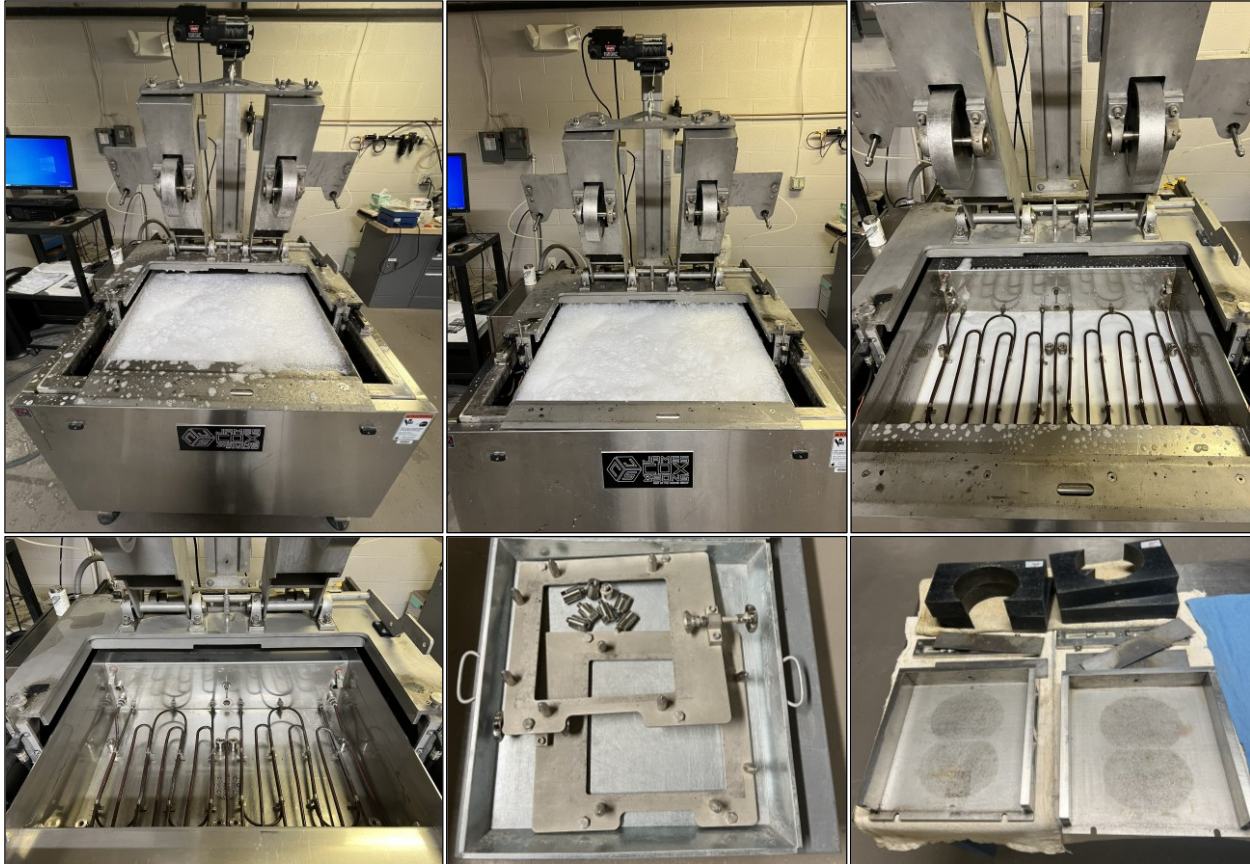


Figure 3: Cleaning process of the HWTT equipment with its fixtures

1  
2  
3 Before HWTT with asphalt pavement samples, the machine was allowed to run for 10 minutes  
4 with empty upside-down molds and other fixtures that were used during a typical test. This was  
5 conducted to obtain a baseline condition for HWTT samples. After completion of the test, the  
6 water solution with the debris was collected using a 10 $\mu$ m filter (Figure 4a and 4b) at the end of  
7 the outlet pipe of the HWTT machine. In addition to the test water that drains naturally, the  
8 machine bath was flashed with room temperature water with a continuous pressure hose for  
9 approximately 10 minutes until the water was cleared. The collected debris and sediment were  
10 immediately transferred to a glass jar along with the filter and sealed (Figure 4c) until it was  
11 characterized for microplastics. The solution collected before the HWTT for unmodified samples  
12 is named 0% LDPE-1 while that for the modified samples is named 6% LDPE-1. The same  
13 procedures were followed to collect sample solutions after the HWTT test with asphalt pavement  
14 samples. The solution collected after the HWTT for unmodified samples is named 0% LDPE-2  
15 while that for the modified samples is named 6% LDPE-2.



Figure 4: Sample collection from (a) HWTT, (b) MiST, and (c) sample transferred to a jar

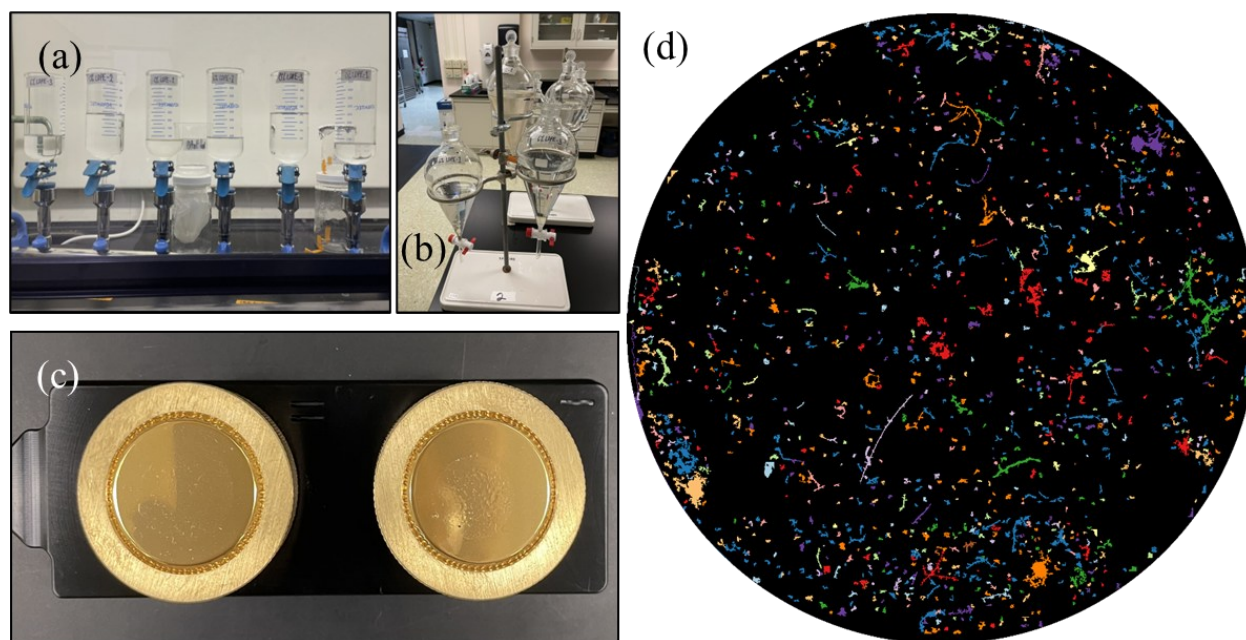
### 2.2.4 Moisture Induced Sensitivity Test

The Moisture-Induced Sensitivity Test (MiST) was conducted following AASHTO TP 140 and ASTM D7870. MiST test subjected compacted asphalt samples to a hydrostatic pore pressure in a confined chamber and high temperature to determine cohesion and adhesion strength of compacted asphalt mixture specimens in aggravated conditions (3500 cycles of 40 psi confined hydrostatic pore pressures at 60 °C, for 20.5 hours in the equipment's pressure chamber). Like HWTT, MiST was not used for its intended purpose but created a condition to induce the release of microplastics from the samples, if any. In addition to intact asphalt pavement samples, the tested HWTT samples that were already damaged were used for MiST in this research. The intact samples are named IC0 for the unmodified and IC6 for the modified conditions. Likewise, the HWTT-damaged unmodified samples evaluated with MiST are called 0% LDPE-3 while that of the modified sample is named 6% LDPE-3. Each MiST subjected two compacted asphalt pavement samples to the mentioned aggravated condition. Sample solution (leachate) from MiST was collected in the same manner as in HWTT (Figure 4b). The 10 $\mu$ m filter was attached at the end of the outlet pipe for debris collection. In addition to the test water, the samples in the MiST equipment were flashed with an extra five gallons of room-temperature water.

### 2.2.5 Microplastic Characterization

After screening samples of the raw materials for microplastics, the following procedures were used to evaluate the collected solutions from mechanical tests. Initially, each sample filter was quantitatively transferred into a respective chemical sample jar using DI water. Then, the sample jar was decanted and extensively rinsed into a separatory funnel for density separation using NaCl for a minimum of 48 hours, and the densest constituents were extracted after this interval. Samples were then quantitatively transferred and filtered through a 25 mm internal diameter, 0.8  $\mu$ m pore, polyester gold coated (PETG) filter membrane. The filter membrane with the particles was carefully transferred and secured to the LDIR sample stage and the stage was inserted into the LDIR instrument and analyzed using a particle analysis method (Microplastic Starter 1.0). For each sample, the LDIR detected and counted the total number of particles present on the PETG with their physical attributes, chemical spectrum, images of the individual particle, and an overall false image of the PETG membrane with deposited particles. Parameters such as Area,

1 Identification, and Quality of the identification for each particle were used for further analysis in  
2 this study. An image of this process with a false image of one of the samples is presented in Figure  
3 5.



4  
5 Figure 5: LDIR samples preparation process (a) and (b) density separation of the particles, (c)  
6 particles on the PETGs, and (d) and false image of one of the samples for this study (Photos  
7 courtesy of PSU-IEE)

### 8 3. Results and Discussions

9 Microplastic characterization using the LDIR built-in library revealed the presence of a wide range  
10 of polymeric and non-polymeric particles across all tested samples, including asphalt raw  
11 materials, background water solutions, and leachates from both modified and unmodified asphalt  
12 specimens. None of the detected particles were positively identified as specific microplastics and  
13 polymeric particle counts remained consistently low. However, their widespread and random  
14 presence across all samples introduced significant complexity in determining whether recycled  
15 RPMA specimens released microplastic.

16 Importantly, the only polymer used in the modified samples was agricultural LDPE, which is the  
17 primary material of interest (Figure 1). While the LDIR did not identify any particles explicitly as  
18 LDPE, it did detect polyethylene (PE) and chlorinated polyethylene (CPE) across both control and  
19 modified samples. These polymers share key spectral similarities with LDPE and are therefore  
20 treated in this study as proxy indicators of LDPE-related particles. For clarity, when using the  
21 built-in LDIR polymer library, PE and CPE are considered particles of interest; when using the  
22 custom-built asphalt library, the relevant particle of interest is labeled LDPE.

23 As with many infrared spectroscopy-based methods, spectral match quality is a continuous metric,  
24 and there is currently no standardized threshold for confirming positive identification of  
25 microplastics in recycled plastic-modified asphalt contexts. Confidence levels are typically  
26 determined at the discretion of the researcher. Any match below the ideal 100% certainty reflects

1 varying degrees of uncertainty. In this study, the following confidence scale was adopted to guide  
2 interpretation:

- 3 • High confidence: Match quality > 95%
- 4 • Medium confidence: Match quality between 90% and 95%
- 5 • Low confidence: Match quality < 90%

6 This confidence framework is consistent with broader scientific practice in polymer identification  
7 but is not universal. Alternative thresholds may be applied depending on analytical goals,  
8 instrumentation, and material complexity.

### 9 **3.1 LDIR Results of the Raw Materials**

10 Table 1 summarizes the relative abundance of polyethylene (PE) and chlorinated polyethylene  
11 (CPE) as a percentage of the total particle count (PPC) in 1 mg of each raw material sample, along  
12 with their corresponding spectral match quality scores. While LDIR detected these polymers  
13 across several samples, it is critical to interpret these results in the context of the method's  
14 limitations.

15 Although LDIR is a powerful technique for rapid microplastic detection and classification, its  
16 effectiveness may be influenced by both the complexity of the material being analyzed and the  
17 structure of the reference library. Materials with broad, overlapping infrared absorption spectra,  
18 such as petroleum-based binders, modifiers, or mineral aggregates, can lead to ambiguous  
19 matches. The LDIR's matching algorithm attempts to assign the best available spectral fit from its  
20 library, which may result in misidentifications when the sample's actual composition is not well-  
21 represented or falls outside the library's spectral content.

22 Table 1: PE and CPE content of the raw materials evaluated with LDIR

Sample of Material (LDIR)	Random mass from bin (g)	Mass for test (mg)	PE PPC (Match Quality) (%)	CPE PPC (Match Quality) (%)
Pan dust (<0.075)	62.1	1.0	0.91 (66)	0.08 (72)
0.075mm	64.4	1.0	1.92 (66)	0.00 (N/A)
0.150mm	119.7	1.0	13.95 (72)	0.00 (N/A)
0.300mm	128.1	1.0	0.00 (N/A)	5.00 (66)
0.600mm	131.2	1.0	3.76 (68)	6.02 (70)
Anti-stripping agent	37.4	1.0	12.12 (66)	3.03 (64)
PG64-22	42.3	1.0	63.6 (93)	0.00 (N/A)

23  
24 As an example, consider the LDIR results for the PE and CPE content in the asphalt binder (PG64-  
25 22), summarized in Table 1. These results are based on microdroplets (~0.016 mm<sup>2</sup> average area)  
26 of binder sprayed onto a glass slide, as described in the raw material screening methodology. Of  
27 the eleven analyzed droplets, LDIR identified seven (64%) as polyethylene (PE), with an average  
28 match quality of approximately 93%. The remaining four droplets (36%) were identified as rubber  
29 with similar match quality scores (not shown in the table).

30 Although a 93% match falls within the "medium confidence" range defined earlier, this  
31 identification is likely a misclassification, a limitation of LDIR's spectral matching algorithm.  
32 LDIR assigns identities by comparing particle spectra to those in its reference library. However,

1 complex, undefined mixtures like asphalt binders are difficult to classify with confidence. PG64-  
2 22 is composed of saturates, aromatics, resins, and asphaltenes, collectively known as SARA  
3 fractions, which do not have fixed molecular structures and instead represent a continuum of  
4 hydrocarbon compounds. Therefore, it does not make sense if the LDIR identifies it as anything.

5 These SARA fractions, especially the saturates and resins, contain long hydrocarbon chains that  
6 produce IR spectra like polyethylene. This spectral overlap likely explains why the binder droplets  
7 were misidentified as PE. Similarly, the resin fraction's structural and functional resemblance (35–  
8 37) to certain rubbers may account for the rubber classification observed in 36% of the  
9 microdroplets.

10 Further characterization methods, such as Raman spectroscopy, pyrolysis-GC/MS, or detailed  
11 SARA fraction analysis, would be required to conclusively validate these identifications.  
12 However, such analyses were beyond the scope of this study.

13 Similarly, for the aggregate samples presented in Table 1, the detected PE and CPE content was  
14 minimal and associated with low match quality and very small particle counts. More than 85% of  
15 the total particles were identified as a diverse mix of non-polymeric materials, including cellulosic  
16 substances, chitin, coal, natural polyamides, calcium carbonate-based particles, and silica. This  
17 composition is consistent with the expected mineral and organic content of aggregates sourced  
18 from limestone quarries and is further supported by ATR results discussed in the following section.

19 Notably, some of the identified substances, such as cellulose and chitin, are among the most  
20 abundant natural organic compounds on Earth. Their presence in the raw materials, and  
21 consequently in the prepared asphalt mixtures, is unsurprising and not considered relevant to the  
22 detection of microplastics in this study. As such, they are not analyzed further.

23 Despite the limitations and high sensitivity of the LDIR method, the detection of microplastics in  
24 raw materials is consistent with a growing body of literature. Numerous studies have documented  
25 microplastics in natural environments, with one recent review calling their presence a threat to  
26 "every sphere of the planet" (38, 39). Microplastics have also been detected in and around mining  
27 operations (40, 41) such as aggregate quarries. In these settings, the presence of microplastics is  
28 often attributed to the abrasion and degradation of polymer-based mining equipment such as tires,  
29 conveyor belts, vehicle parts, storage containers, and ventilation systems. Additionally, airborne  
30 and waterborne transport can introduce microplastics from external sources. In the context of this  
31 study, where aggregates were sourced from a limestone quarry, the detection of PE, CPE, and other  
32 polymeric particles is therefore not unexpected. For example, after passing through crushers, the  
33 aggregates are conveyed via long polymer-based belts to storage piles. These belts frequently wear  
34 down and are replaced, shedding fine polymeric debris in the process. Even if such material  
35 comprises a small fraction of the total mined volume or in trace amounts, they will still be detected  
36 in an LDIR characterization.

### 37 **3.2 ATR Results of the Raw Materials**

38 Table 2 presents the ATR results for the aggregate samples with the highest spectral match quality.  
39 In all cases, the aggregates were identified as polysulfide caulking and high calcium carbonate  
40 (CaCO<sub>3</sub>) filler with a spectral match quality exceeding 80%. Although the aggregates are not  
41 polysulfide caulking material, the principal component of both polysulfide caulking and the  
42 limestone (e.g., aggregates used for this research) are CaCO<sub>3</sub>. Hence, the ATR device matches the  
43 aggregates to the closest available polymer in its library, in this case, polysulfide caulking.

1 Table 2: ATR results for the raw materials

Sample of Material (ATR)	IR Spectra Best Match (ATR Library)	Match Quality (%)
Pan dust (<0.075)	Polysulfide Caulking, high CaCO <sub>3</sub> filler	86.638
0.075mm	Polysulfide Caulking, high CaCO <sub>3</sub> filler	89.798
0.150mm	Polysulfide Caulking, high CaCO <sub>3</sub> filler	90.297
0.300mm	Polysulfide Caulking, high CaCO <sub>3</sub> filler	90.312
0.600mm	Polysulfide Caulking, high CaCO <sub>3</sub> filler	88.151
1.180mm	Polysulfide Caulking, high CaCO <sub>3</sub> filler	90.011
2.360mm	Calcium Carbonates (CaCO <sub>3</sub> )	84.943
4.750mm	Polysulfide Caulking, high CaCO <sub>3</sub> filler	83.935
9.500mm	Polysulfide Caulking, high CaCO <sub>3</sub> filler	85.304

2

3 **3.3 LDIR Results of the Particles Collected from the Solutions Before the HWTT**

4 LDIR analysis of the water solution samples collected prior to the Hamburg Wheel Tracking Test  
5 (HWTT) (0% LDPE-1 and 6% LDPE-1) revealed the presence of various polymeric and non-  
6 polymeric particles. Figures 6a and 7a display the results based on the LDIR's built-in polymer  
7 library, while Figures 6b and 7b present the same samples analyzed with the custom asphalt library.

8 Across all four analyses, several types of particles were detected, including those chemically  
9 similar to the target polymers (PE, CPE, and LDPE). However, the identifications were generally  
10 of low confidence and represented a small proportion of the total particle count. While the sample  
11 labels reference LDPE content (0% and 6%), it is important to clarify that these solutions were  
12 collected before HWTT of specimens with 0% and 6% LDPE content, respectively. The solutions  
13 did not contain added LDPE themselves. Furthermore, the two solutions were collected on  
14 different days under independent conditions.

15 In Figures 6a–7b, particle identification quality is represented by a dot-whisker plot on the left y-  
16 axis (mean  $\pm$  standard deviation), and the relative abundance of each unique particle type is shown  
17 as a percentage of total particle count (PPC) on the right y-axis. Particles of interest (PE, CPE, and  
18 LDPE) are highlighted and labeled for ease of reference.

19 The consistent detection of PE and CPE in both pre-test samples suggests the influence of  
20 background contamination, which may originate from the HWTT apparatus itself. Components  
21 such as plastic molds (often made from high-density polyethylene, HDPE) and piping (commonly  
22 LDPE) are potential sources of leached or abraded particles. Despite thorough cleaning between  
23 tests, residual contamination is plausible, particularly given that the HWTT equipment had  
24 previously been used for RPMA testing involving the same type of agricultural LDPE. In addition,  
25 the shared laboratory environment, where various recycled plastics are stored and handled, further  
26 increases the likelihood of airborne or surface-borne contamination, which could have affected  
27 both equipment and other materials in the lab.

28 While background solutions were collected before each HWTT and the suspended particles were  
29 characterized for microplastics, their values were not meant to be subtracted from post-test  
30 measurements due to uncontrolled variations in lab use, equipment exposure in between these  
31 solutions collection, and timing of the collection. Instead, these data are used qualitatively to show  
32 the pervasive presence of microplastic contamination across this testing environment.

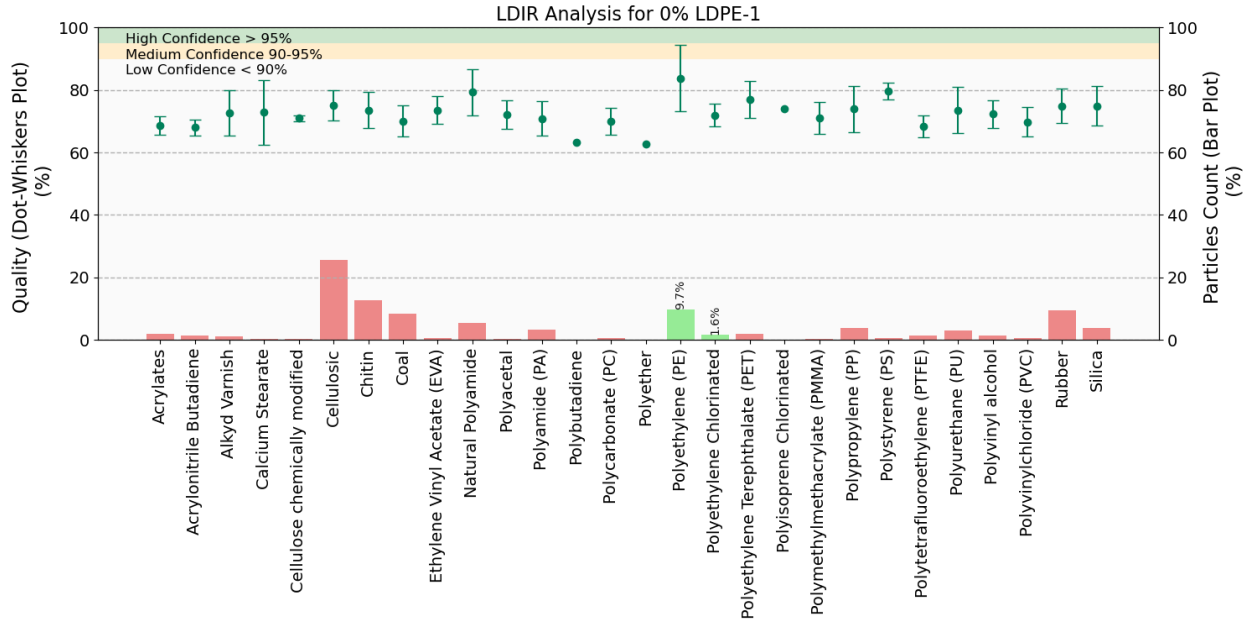


Figure 6a: LDIR analysis results for 0% LDPE-1 (built-in library)

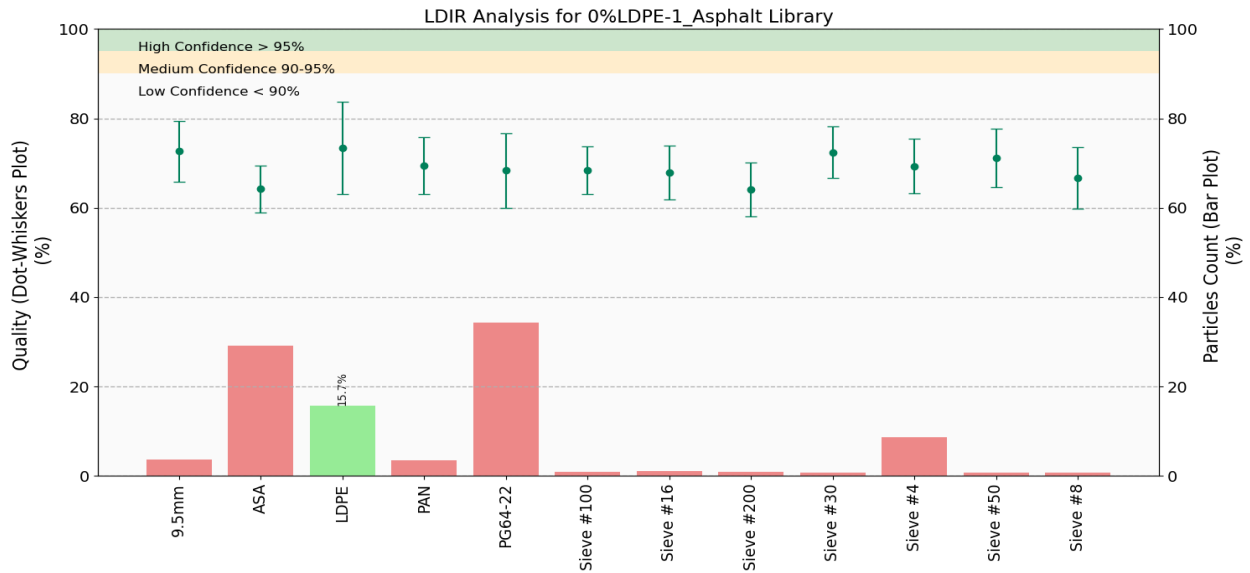


Figure 6b: LDIR analysis results for 0%LDPE-1 (custom-built asphalt library)

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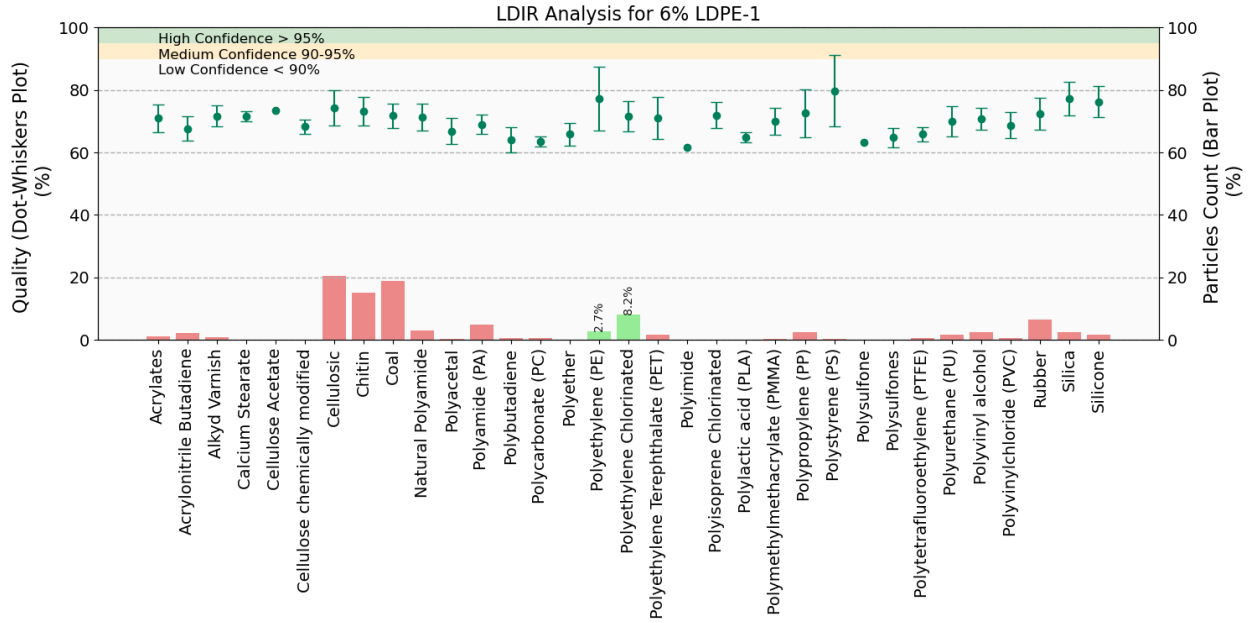


Figure 7a: LDIR analysis results for 6% LDPE-1 (built-in library)

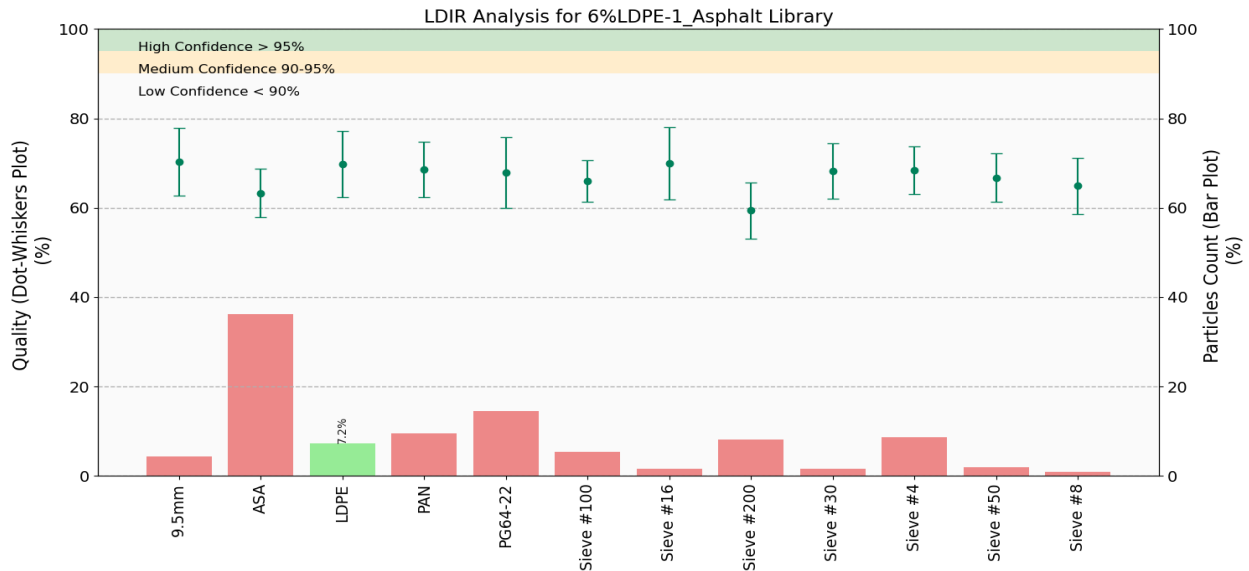


Figure 7b: LDIR analysis results for 6%LDPE-1 (custom-built asphalt library)

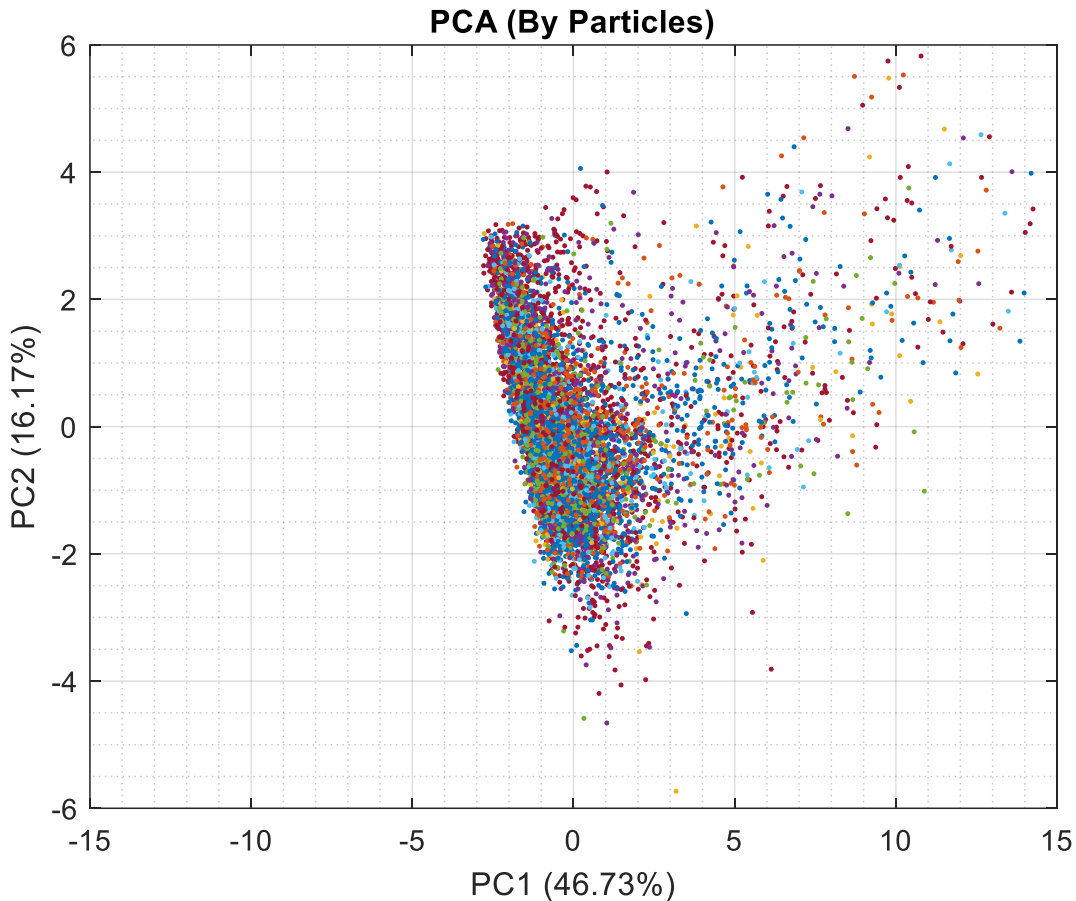
### 3.4 LDIR Results of the Particles from the Asphalt Pavement Samples

Given the wide range of particles detected in both the raw materials and background solutions, several analytical strategies were employed to assess whether recycled plastic-modified asphalt samples released more polymeric particles, particularly microplastics, than unmodified controls, irrespective of the particles of interest.

An initial exploratory data analysis was conducted using the physical attributes of particles detected via the LDIR using its built-in library. These attributes included width, height, diameter, aspect ratio, area, perimeter, eccentricity, circularity, and solidity. However, no single variable showed a consistent or distinguishable pattern between modified and unmodified samples.

1 Hence, to explore whether combinations of these features might reveal underlying structure,  
 2 Principal Component Analysis (PCA), a commonly used linear dimensionality reduction  
 3 technique, was applied. The first round of PCA grouped all particles across the six test conditions  
 4 according to their material identification (Figure 8). The goal was to determine whether particles  
 5 linked to plastic modification, particularly those identified as PE or CPE, exhibited distinct  
 6 clustering behavior.

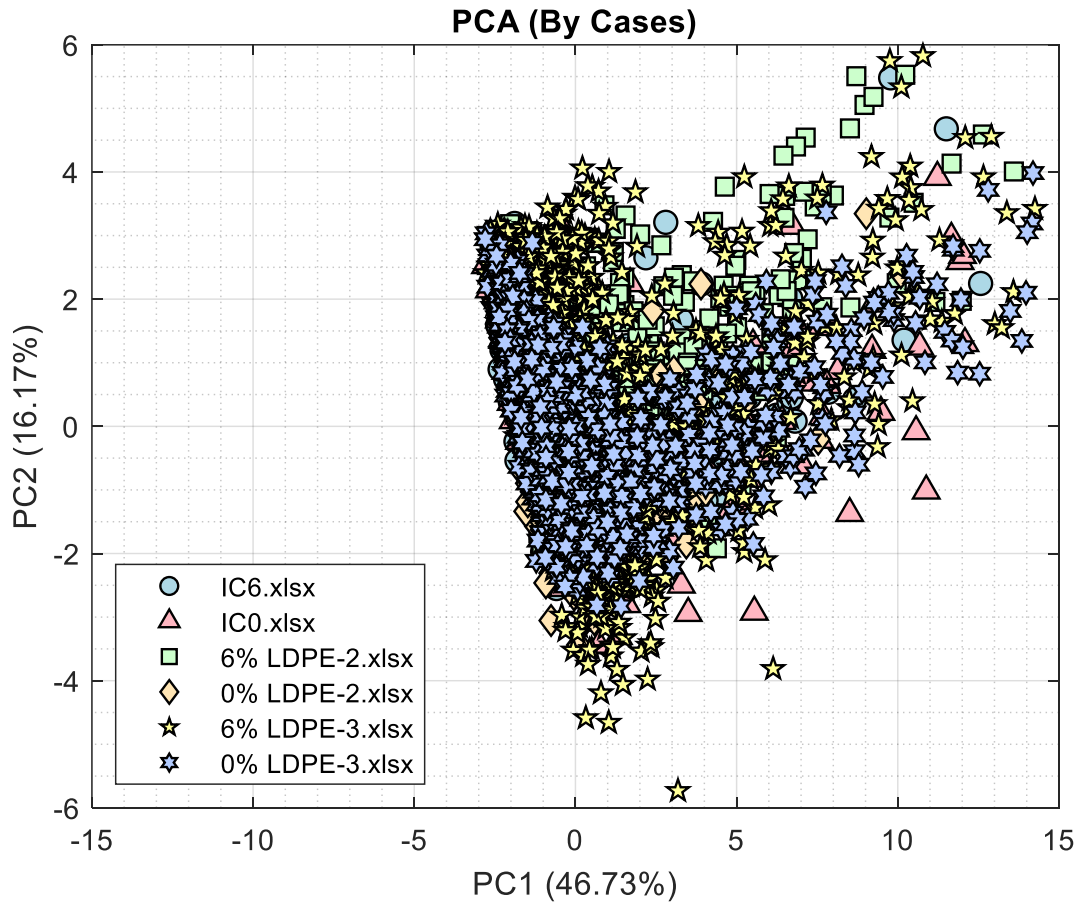
7 Although both PE and CPE were detected across all test cases, the PCA revealed no consistent  
 8 separation or clustering based on identification type alone, suggesting that individual particle  
 9 classifications were insufficient for distinguishing modification conditions.



10  
 11 Figure 8: PCA by particles type (legend is not shown as it includes ~ 35 unique material)

12 PCA was then applied based on the six distinct sample cases (Figure 9). Although some visual  
 13 dispersion and partial grouping were observed, the results did not reveal any definitive clustering  
 14 patterns that could reliably distinguish the samples by case.

15 It should be noted that, while other data science techniques, such as nonlinear dimensionality  
 16 reduction or machine learning-based clustering could be applied that might uncover subtle  
 17 patterns, they were not pursued in this study, as the primary objective was to assess observable  
 18 differences under realistic laboratory conditions using interpretable, conventional methods.



1  
2 Figure 9: PCA by case (IC6, IC0, 6% LDPE-2, 0% LDPE-2, 6% LDPE-3, and 0% LDPE-3)

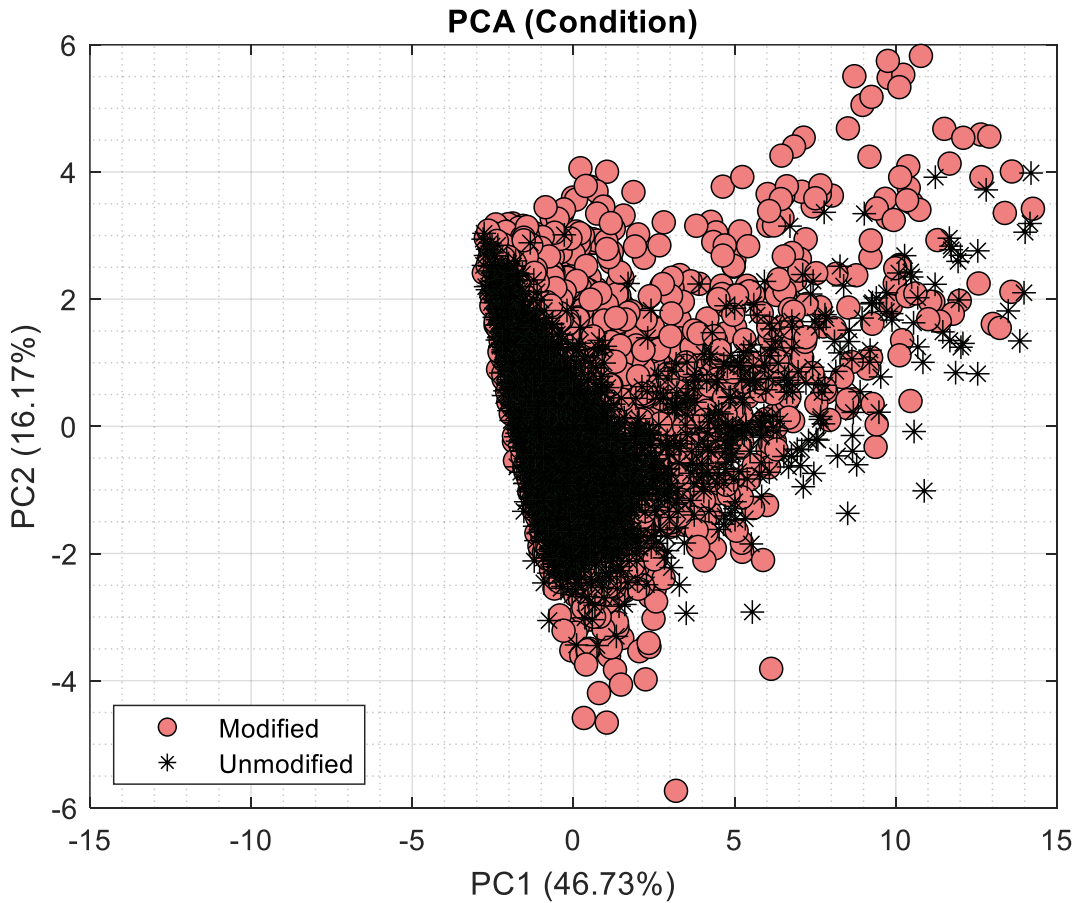
3 Next, PCA was conducted by grouping particles according to modification conditions, either  
4 modified or unmodified, as shown in Figure 10. This approach produced a clearer clustering  
5 pattern: the unmodified samples formed a relatively compact cluster, while the modified samples  
6 exhibited greater dispersion, spatially surrounding the unmodified group.

7 This behavior aligns with the material composition. The unmodified set, denoted as:

8  $\mathbf{u} = \{\text{aggregates, binder, anti-stripping agent}\}$ , which represents the base mix, while the  
9 modified set can be expressed as:

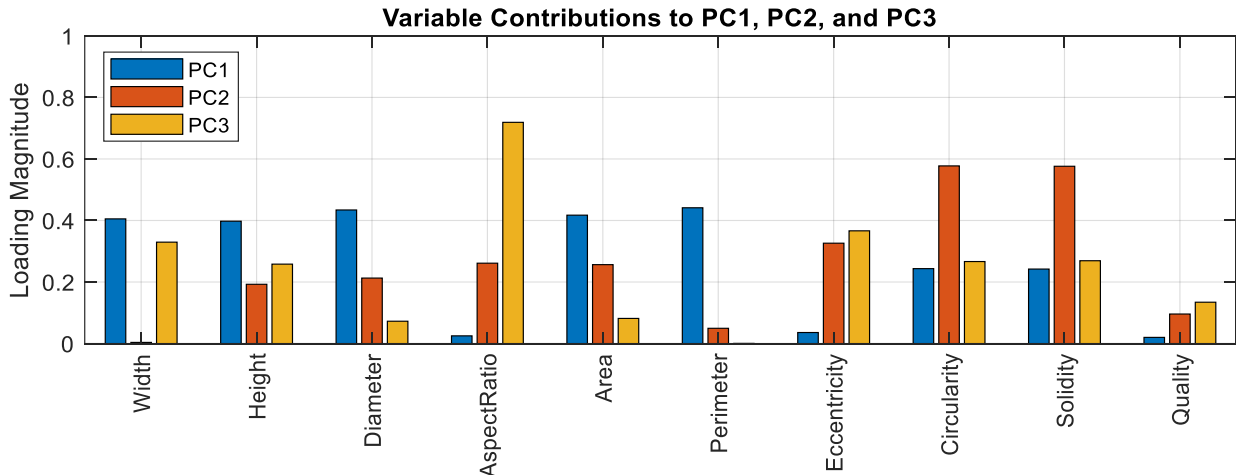
10  $\mathbf{m} = \mathbf{u} \cup \{\text{LDPE particles}\}.$

11 Both groups share the same foundational materials, but the inclusion of LDPE in the modified  
12 samples introduces additional particle types and morphological variability, leading to broader  
13 feature distributions in the PCA space.



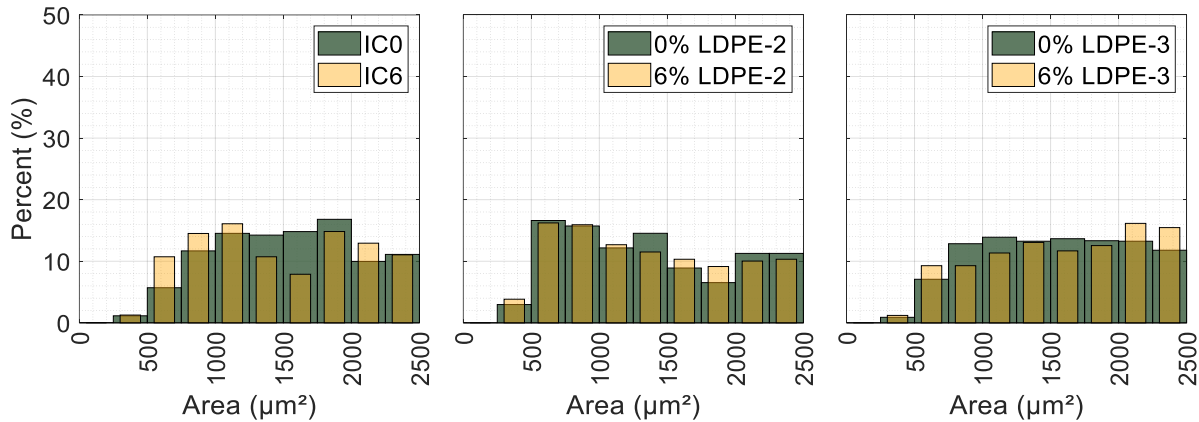
1  
2 Figure 10: PCA by modification condition (modified vs unmodified)

3 Since PCA revealed a distinct separation between modified and unmodified samples, a loading  
4 analysis was conducted to identify which particle attributes contributed most to the principal  
5 components (Figure 11). The analysis showed that Principal Component 1 (PC1) was dominated  
6 by size-related variables, including area, width, height, diameter, and perimeter, while Principal  
7 Component 2 (PC2) was primarily influenced by shape-related characteristics, such as solidity,  
8 circularity, and eccentricity.

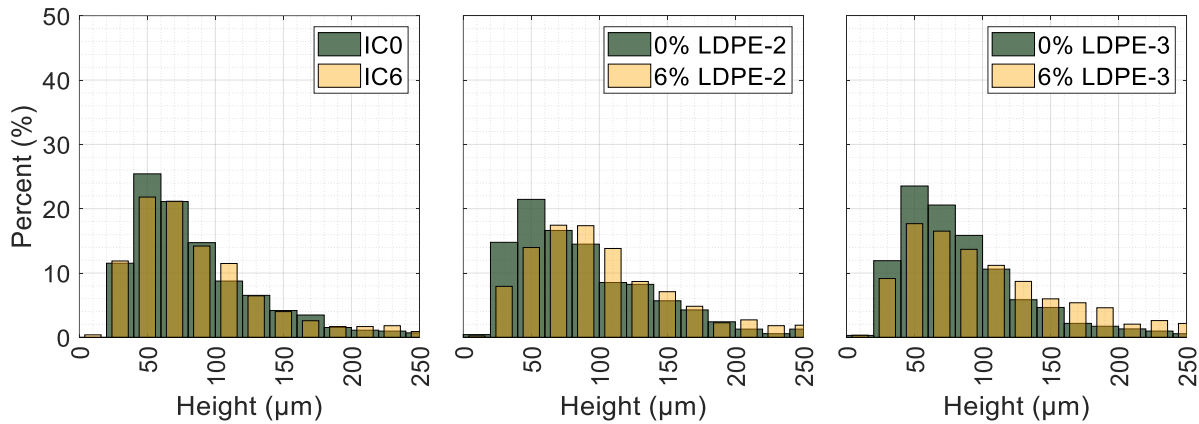


9

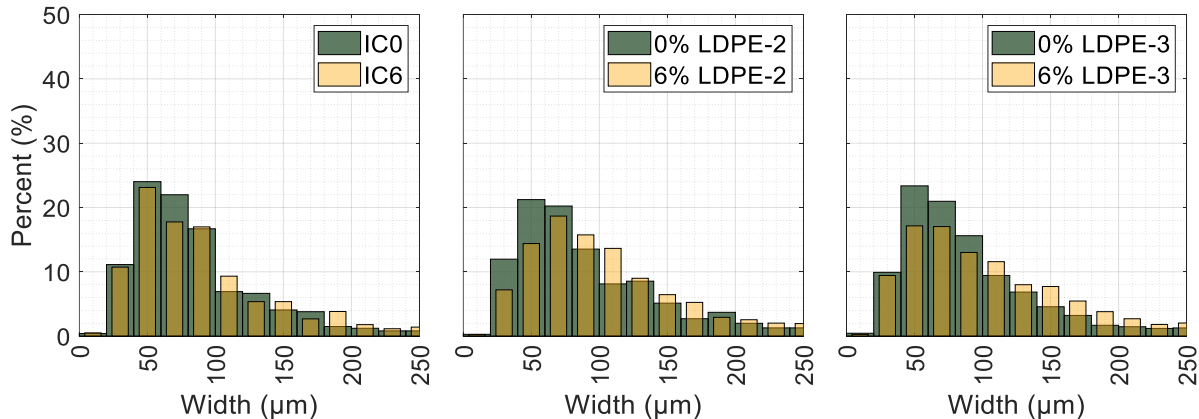
1 Figure 11: PCA loading contribution of each variable to the first three principal components  
 2 Consequently, size-related parameters of the detected particles were selected for further analysis.  
 3 Distributions of these parameters, grouped by modification conditions, are shown in Figures 12  
 4 through 16. While all size-related variables contributed comparably to PC1, particle area was  
 5 chosen as the primary variable for quantitative comparison in this study, due to its broad  
 6 representativeness of particle size. That said, the same analytical framework could be applied using  
 7 other features, particularly when particle shape (e.g., fibers, pellets, spheres, or fragments) is of  
 8 specific interest for classification or environmental behavior modeling.



9  
10 Figure 12: Area distribution of the particles from different cases

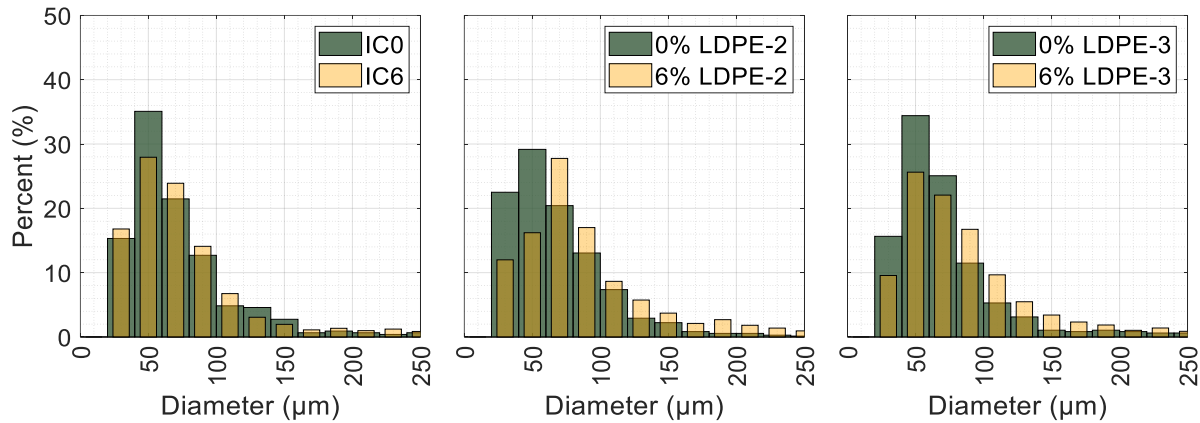


11  
12 Figure 13: Characteristic Size (Height) distribution of the particles from different cases



1

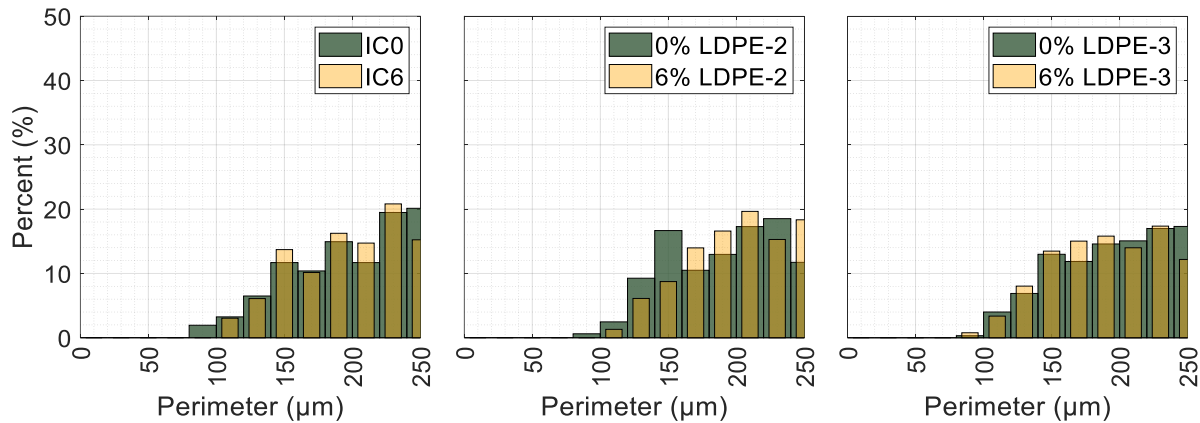
Figure 14: Width distribution of the particles from different cases



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Figure 15: Diameter distribution of the particles from different cases



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Figure 16: Perimeter distribution of the particles from different cases

6 To quantitatively compare the release potential of microplastic-like particles across different  
 7 samples, a Normalized Area Index (NAI) was developed. For each test case, the total area of  
 8 detected particles was normalized by the mass of the corresponding asphalt pavement specimen,  
 9 resulting in a metric expressed in  $\mu\text{m}^2/\text{g}$ . This allowed for a standardized comparison of particle  
 10 load between samples with differing test masses.

11 The NAI is defined as:

12 
$$NAI = \frac{\sum A}{m}$$

13 where:

14 *A* is the sum of area of all the particles for a sample in  $\mu\text{m}^2$

15 *m* is the total mass of the corresponding asphalt pavement sample in grams

16 Higher NAI values may indicate an increased potential for the release of microplastics or related  
 17 micro polymer particles. While the metric captures all detected particles, subsequent interpretation  
 18 in this study focuses specifically on the particles of interest (PE, CPE, and LDPE).

1 Sample mass values used for normalization are provided in Table 3. The two water solution  
 2 samples (0% LDPE-1 and 6% LDPE-1) were excluded from this analysis, as they were not  
 3 associated with measured sample masses and were collected prior to HWTT exposure.

4 Table 3: Mass and number of particles for each case

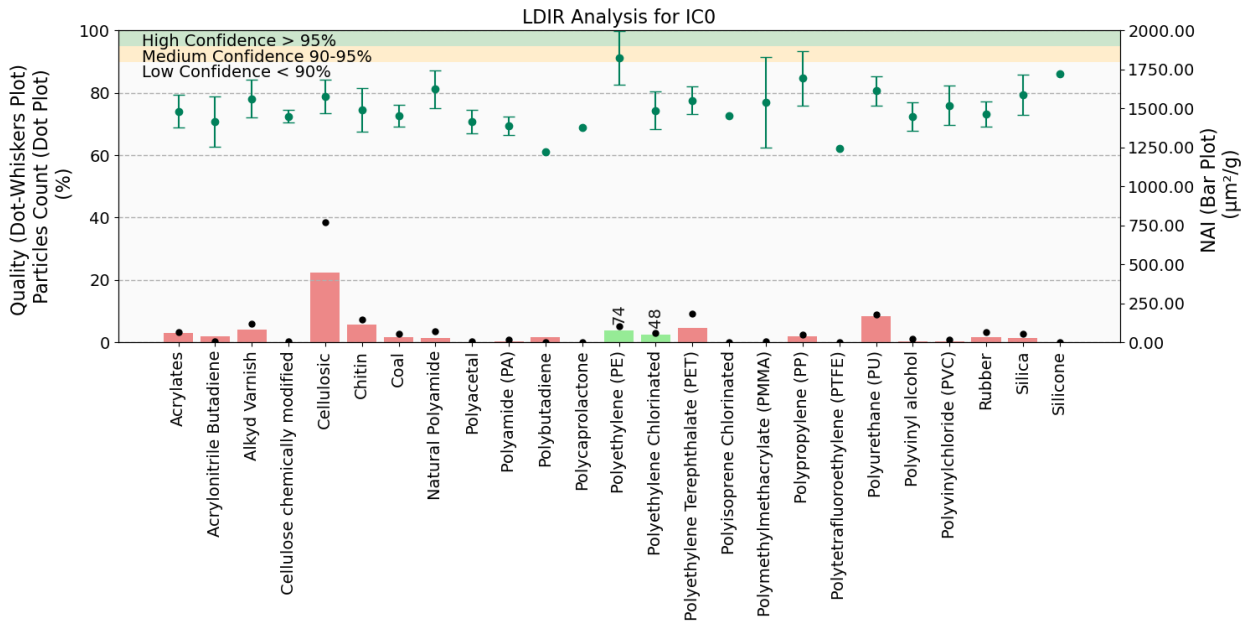
Specimen	Type of Specimens	No. of Specimens	Total Mass (g)	No. of Particles
IC0	Uncut Disks	2	5033.3	796
IC6	Uncut Disks	2	5037.7	831
0% LDPE-2	HWTT	4	9750.30	723
6% LDPE-2	HWTT	4	9760.60	1433
0% LDPE-3	HWTT and MiST	2	4848.7	2847
6% LDPE-3	HWTT and MiST	2	4869.3	2029

5

6 **3.4.1 LDIR Results of the Particles from the Intact Condition MiST Test**

7 The NAI values for samples collected after the Intact Condition MiST test (IC0 and IC6) are shown  
 8 in Figures 17a and 18a (based on the LDIR built-in library) and Figures 17b and 18b (based on the  
 9 custom asphalt library). While the primary discussion in this section focuses on the built-in library  
 10 results for consistency and broader comparability, the custom library results are included to  
 11 provide a complete representation of the data.

12 In these figures, the NAI values of individual particle types, including the highlighted particles of  
 13 interest (PE, CPE, and LDPE, shown in bright green), are plotted on the right y-axis. The left y-  
 14 axis displays both the mean identification quality ( $\pm$  standard deviation) and the percentage of each  
 15 particle type relative to the total particle count for that case, with both metrics expressed as  
 16 percentages.



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Figure 17a: LDIR analysis results for IC0 (built-in library)

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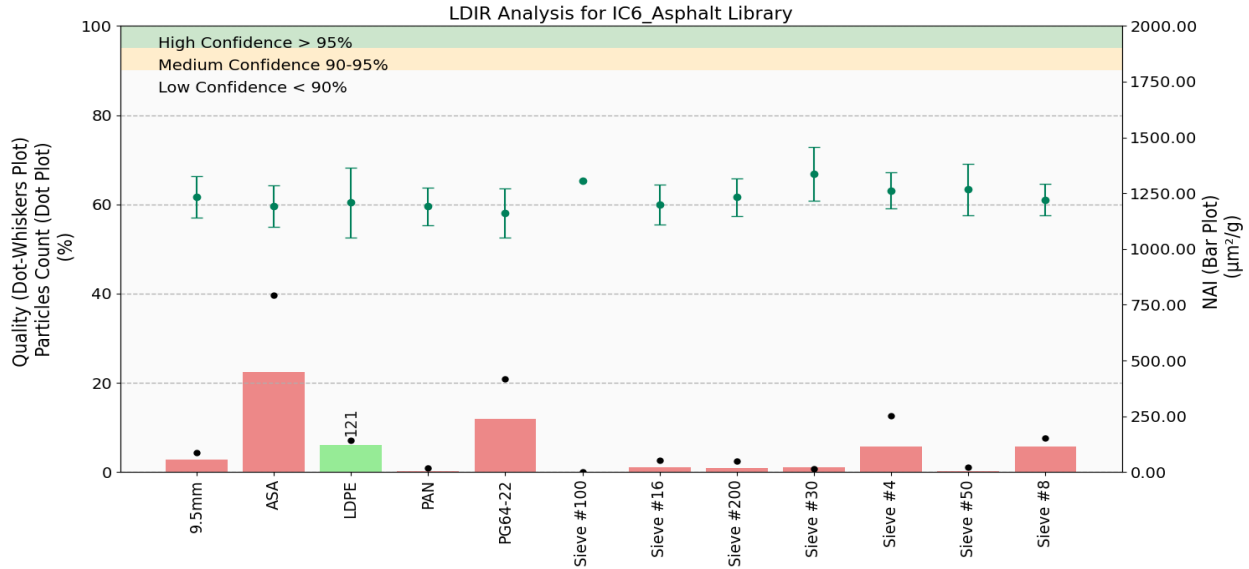


Figure 18b: LDIR analysis results for IC6 (custom built asphalt library)

Comparing IC0 and IC6, it is observed that despite having nearly identical sample masses (5033.3 g vs. 5037.7 g) and total particle counts (796 vs. 831), the normalized area index (NAI) for the particles of interest, PE and CPE, is higher in the unmodified sample (IC0) than in the modified sample (IC6). While this may initially seem counterintuitive, it likely results from a combination of factors.

First, as shown in Table 1, the asphalt binder is often identified by LDIR as PE with high match quality, due to overlapping IR spectra. Therefore, it is expected that both IC0 and IC6 which contain the same binder, would show strong PE-like signals. However, the classification dynamics may shift in IC6 due to the addition of actual LDPE particles, which the LDIR may also interpret as PE. In this case, the instrument may be attributing PE identifications in IC6 to LDPE particles rather than to binder (closer spectral match between LDPE and PE compared to PG64-22 and PE), which could reduce the number of PE-classified particles overall, especially given the low dosage of LDPE (6% of the 5.9% binder content). In contrast, IC0 contains no LDPE, so a higher fraction of particles may be classified as PE due solely to binder exposure.

Second, a potential coating or encapsulation effect may also influence the results. In IC6, LDPE particles might be entirely or partially coated with binder during mixing. Since LDIR identifies materials based on their surface infrared characteristics, the system may interpret these coated particles as binder, or binder-like substances, instead of LDPE or PE.

Finally, particle morphology may also play a role. As shown in Figure 12, the IC0 sample contains a slightly greater proportion of larger-area particles compared to IC6. Since the NAI is area-based, this could further elevate the normalized values for IC0, despite both samples having comparable particle counts.

### 3.4.2 LDIR Results of the Particles from the Damaged Condition MiST Test

The NAI values for samples tested under damaged conditions, that is, MiST testing conducted after HWTT exposure (0% LDPE-3 and 6% LDPE-3), are shown in Figures 20a and 21a for the built-in library analysis, and Figures 20b and 21b for the custom asphalt library.



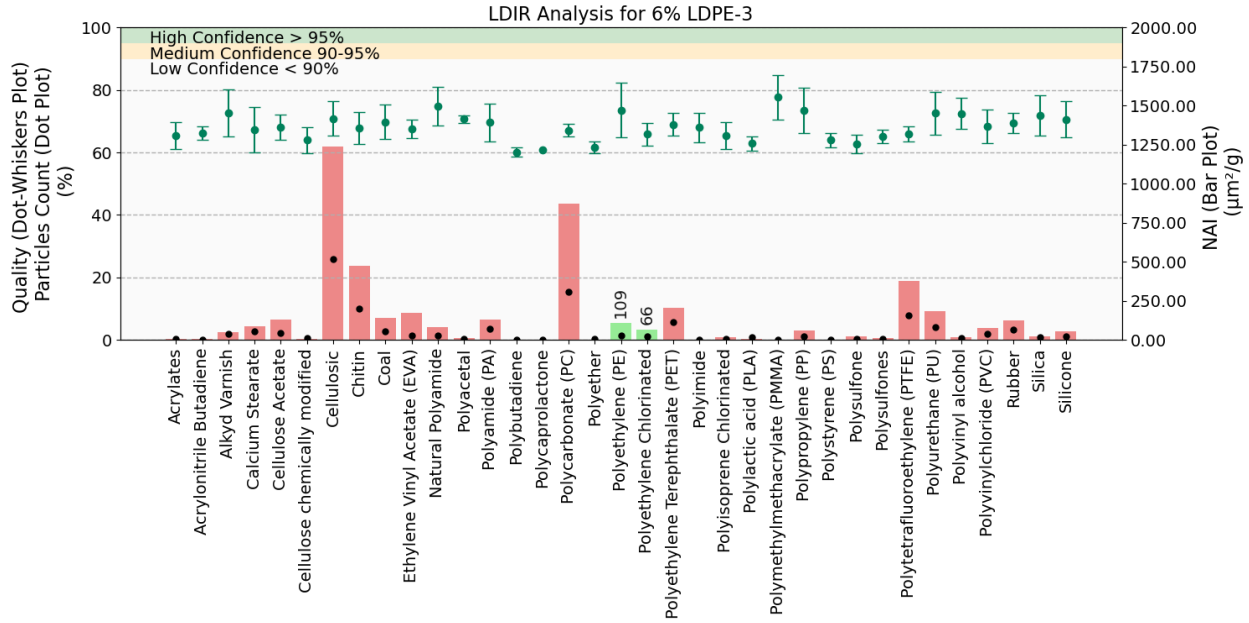


Figure 21a: LDIR analysis results for 6% LDPE-3 (built-in library)

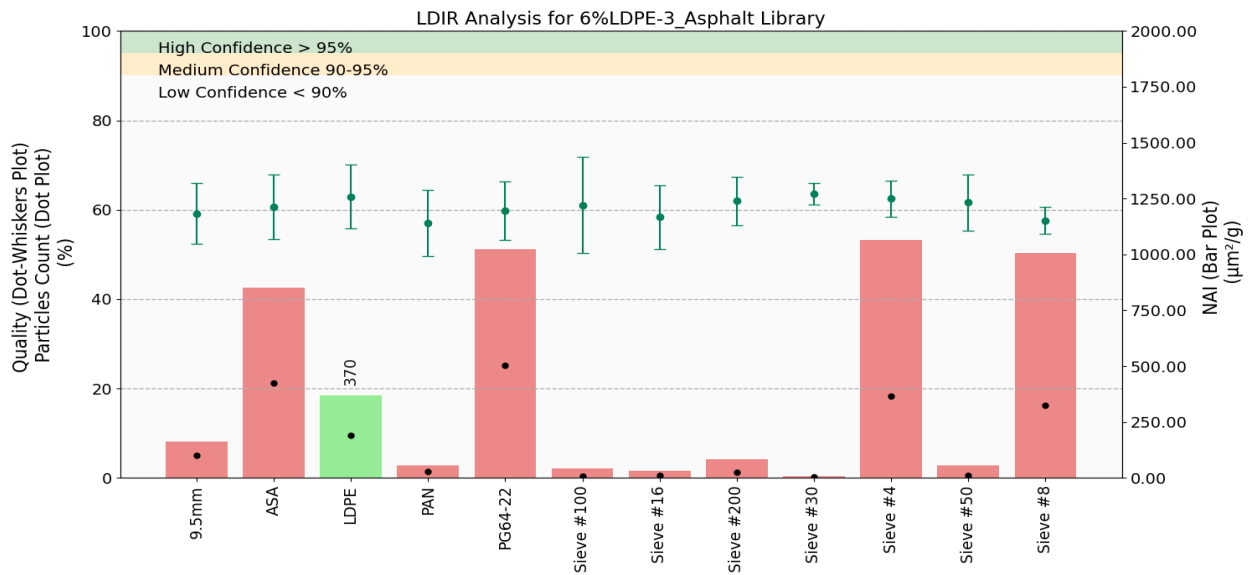


Figure 21b: LDIR analysis results for 6% LDPE-3 (custom-built asphalt library)

### 3.4.3 LDIR Results of the Particles from HWTT

The NAI values for samples collected after Hamburg Wheel Tracking Test (0% LDPE-2 and 6% LDPE-2) are shown in Figures 22a and 23a for the built-in library and in Figures 22b and 23b for the custom asphalt library.

Comparing these cases, both contain particles of interest, but unlike the results from the MiST tests, the modified sample (6% LDPE-2) exhibited higher NAI values than the unmodified sample. This suggests that under the specific mechanical loading and water exposure conditions of the HWTT, the inclusion of LDPE may result in greater particle shedding or at least higher detection of LDPE-like fragments.

1 One possible explanation is that HWTT's repeated wheel loading and immersion may create  
 2 surface wear or abrasion patterns that preferentially affect LDPE domains in the modified asphalt.  
 3 Unlike MiST, which uses cyclic hydrostatic pressure with limited mechanical contact, HWTT  
 4 involves direct physical deformation, which may fragment LDPE into more detectable particles.  
 5 This could account for the increased presence of PE/CPE in the modified sample after HWTT.  
 6 Moreover, unlike the MiST tests, where binder-related misclassification may obscure differences  
 7 between samples, the harsher mechanical damage in HWTT may have exposed or dislodged LDPE  
 8 particles more directly, leading to stronger signal attribution in LDIR.  
 9 That said, the number and area of particles of interest remain relatively low in absolute terms, and  
 10 the match confidence scores are still below the threshold for definitive material identification.  
 11 These findings should therefore be interpreted with caution and within the broader context of  
 12 background contamination and material complexity discussed previously.

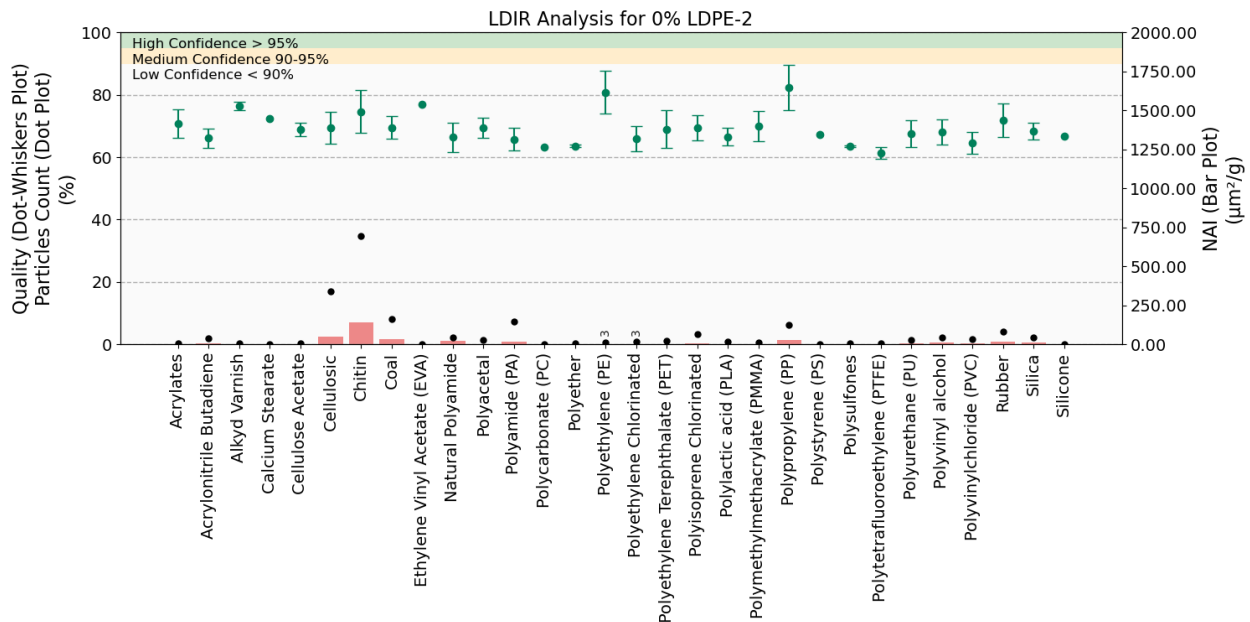


Figure 22a: LDIR analysis results for 0% LDPE-2 (built-in library)

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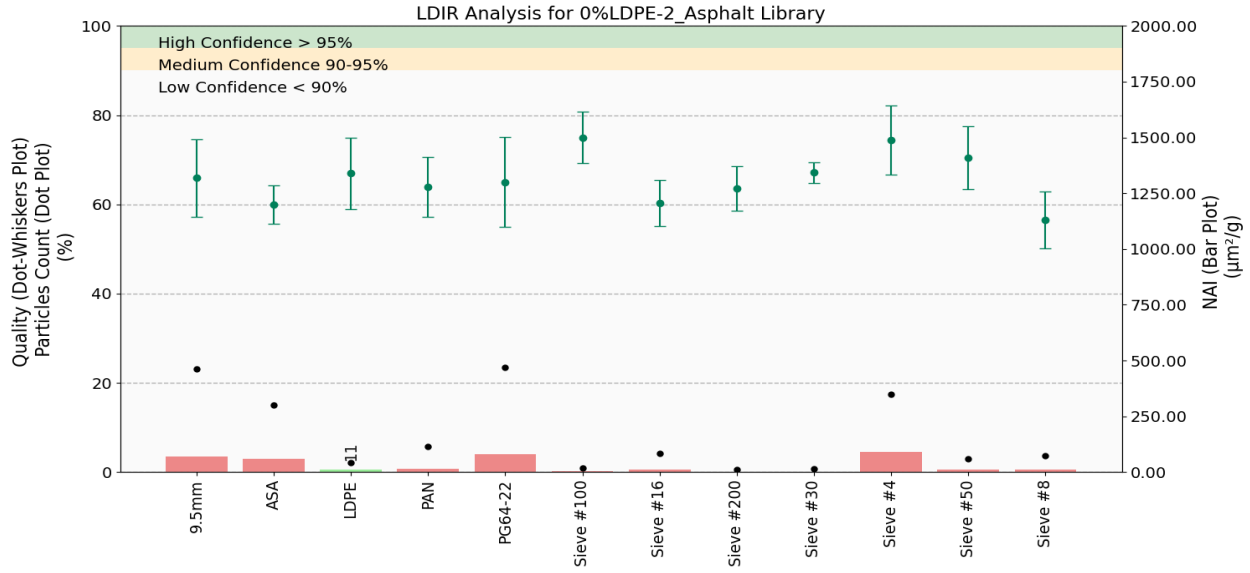


Figure 22b: LDIR analysis results for 0% LDPE-2 (custom-built asphalt library)

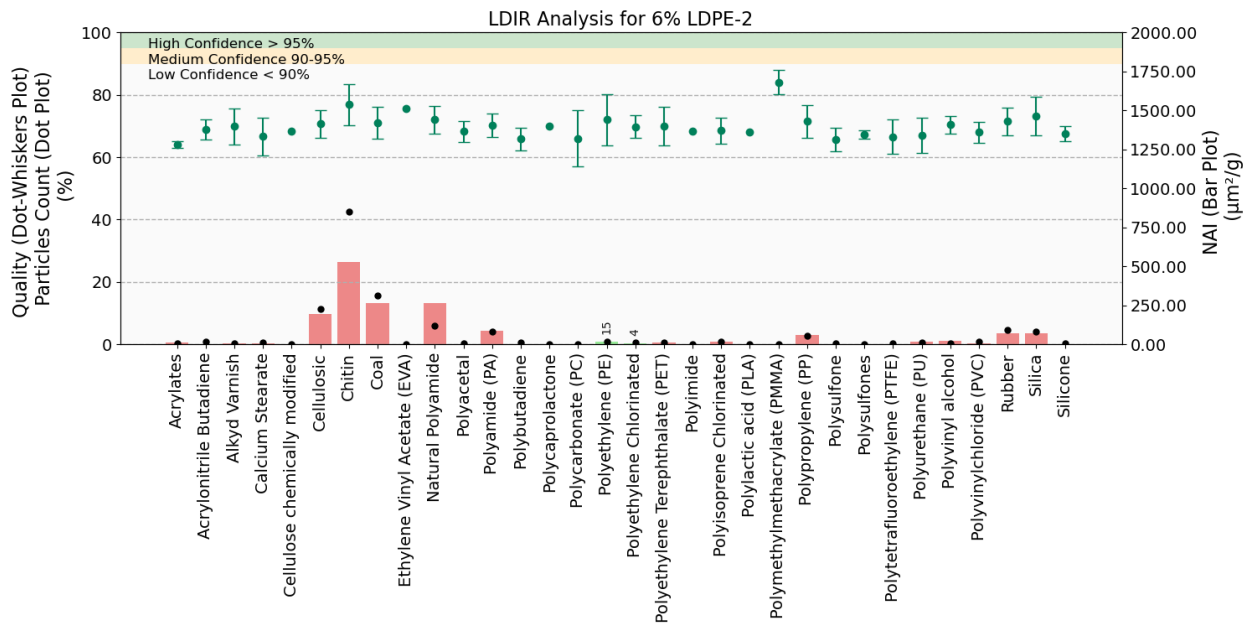


Figure 23a: LDIR analysis results for 6% LDPE-2 (built-in library)

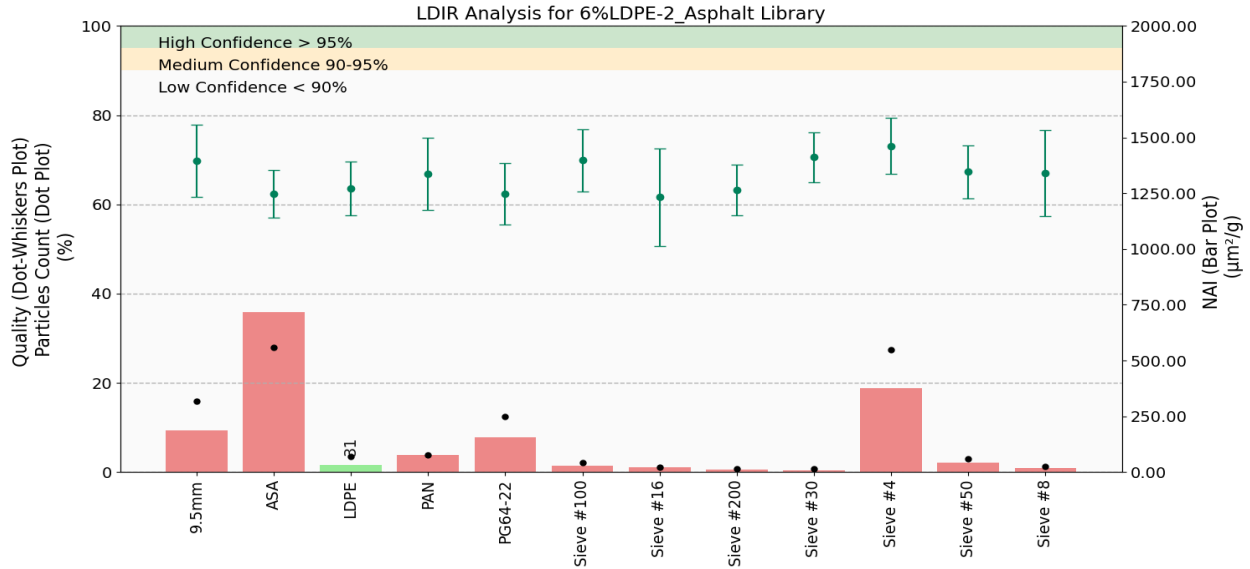


Figure 23b: LDIR analysis results for 6% LDPE-2 (custom-built asphalt library)

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3 Furthermore, it is important to note that the comparisons above between modified and unmodified  
4 samples were based exclusively on the particles of interest (PE, CPE, and LDPE). However, given  
5 the pervasive background contamination observed throughout the study, affecting raw materials,  
6 equipment, and even pre-test solutions, it was necessary to extend the analysis to include all  
7 detected particles, both polymeric and non-polymeric. Hence, when the Normalized Area Index  
8 (NAI) is recalculated on this broader basis, the modified samples consistently exhibit equal or  
9 higher NAI values across test conditions, as shown in Figure 24. This trend is also supported by  
10 the Principal Component Analysis, which revealed greater dispersion of particle features in the  
11 modified samples relative to the unmodified ones.

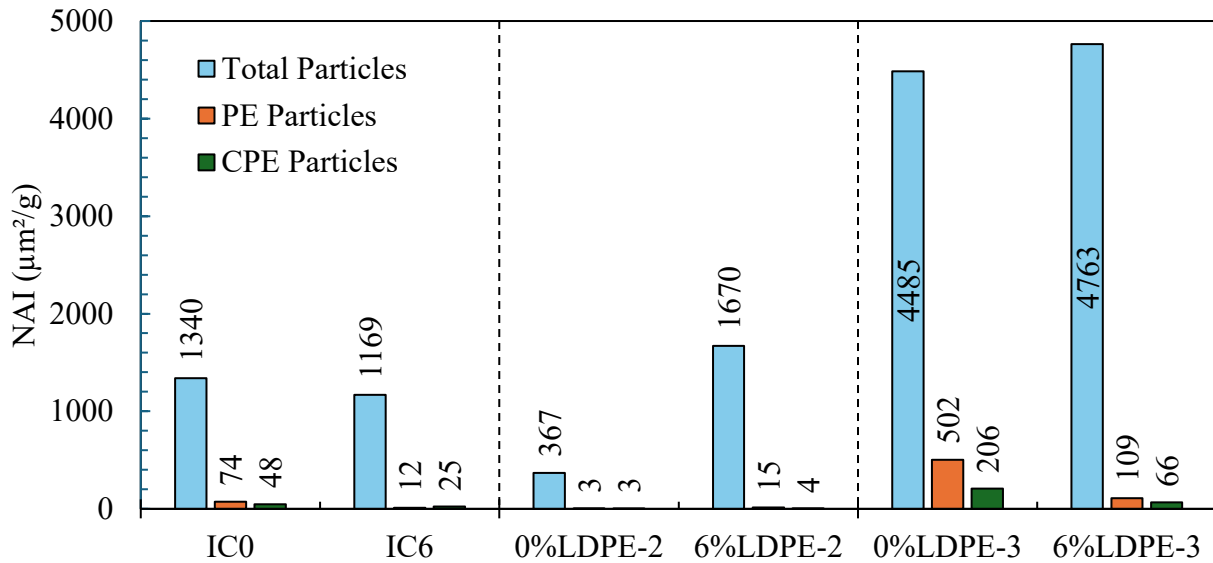


Figure 24: NAI values based on total particles compared with particles of interest for each case

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1 **4. Conclusions and Recommendations**

2 This study investigated the potential release of microplastic particles from Recycled Plastic  
3 Modified Asphalt (RPMA) using agricultural Low-Density Polyethylene (LDPE) film as a  
4 modifier. Modified and unmodified asphalt pavement samples were subjected to Hamburg Wheel  
5 Tracking Test (HWTT) and Moisture Induced Sensitivity Tester (MiST), with leachate samples  
6 analyzed using Laser Direct Infrared Imaging (LDIR) and Attenuated Total Reflectance (ATR).  
7 Raw materials and background solutions were also screened to establish a baseline and develop a  
8 custom asphalt spectral library.

9 Across all samples, including raw materials, background solutions, and unmodified controls, a  
10 wide range of polymeric and non-polymeric particles were detected. PE and CPE particles, which  
11 share spectral similarities with LDPE, were found inconsistent in both modified and unmodified  
12 samples, often at low particle counts and with match quality below thresholds for definitive  
13 identification. Notably, the LDIR system did not positively identify any particles as LDPE.

14 The pervasive detection of these particles, even in unmodified samples and background water,  
15 underscores a broader challenge in microplastic research where contamination appears ubiquitous,  
16 even in controlled laboratory settings. This contamination, coupled with the spectral similarity  
17 between various particles, complicated the efforts to isolate the specific contribution of recycled  
18 LDPE to microplastic release.

19 While some patterns emerged, these results should be interpreted with caution. Limitations in  
20 spectral resolution, overlapping material signatures, and the absence of more definitive chemical  
21 identification techniques (e.g., pyrolysis-GC/MS or Raman spectroscopy) prevented conclusive  
22 attribution of microplastic release to the add LDPE modifier.

23 Ultimately, the study highlights both the analytical potential and the interpretive limitations of  
24 current microplastic detection tools when applied to complex construction materials such as  
25 asphalt pavement. Future research should aim to refine analytical protocols, control background  
26 contamination more rigorously, and incorporate complementary techniques to validate  
27 microplastic presence and identity with higher confidence.

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1 **Artificial Intelligence Disclaimer**

2 This manuscript was edited and subsequently ai-peer reviewed using OpenAI’s ChatGPT, using  
3 the following prompt designed to emulate a rigorous, multi-perspective scholarly review. The AI-  
4 generated feedback was incorporated by the author, and this document reflects the final revised  
5 version.

6

7 **The Prompt:**

8 You are assigned to conduct a comprehensive academic peer review of the attached manuscript,  
9 emulating the role of three independent expert reviewers. Each reviewer should assess the paper  
10 from a distinct disciplinary perspective:

11 • **Reviewer #1 – Materials Science**

12 A researcher with a broad background in materials science, evaluating the manuscript for  
13 technical rigor, experimental clarity, novelty, and contribution to the discipline.

14 • **Reviewer #2 – Recycled Plastics Modified Asphalt Pavement (RPMA)**

15 A domain specialist assessing methodological soundness, relevance of findings to  
16 pavement engineering, and alignment with existing knowledge and industry practices.

17 • **Reviewer #3 – Environmental Sustainability & Microplastics**

18 An expert focused on environmental risk, sustainability, and microplastic behavior within  
19 infrastructure systems. This reviewer emphasizes unintended consequences and lifecycle  
20 impacts.

21 Each reviewer was instructed to:

22 1. Evaluate the manuscript according to established peer review criteria, including:

- 23 • Originality and significance
- 24 • Clarity of objectives and coherence of argument
- 25 • Methodological appropriateness and transparency
- 26 • Quality of results and interpretation
- 27 • Engagement with literature
- 28 • Validity of conclusions
- 29 • Societal and ethical implications

30 2. Identify strengths and weaknesses constructively, and provide specific, actionable  
31 recommendations for improvement.

32 3. Maintain a formal academic tone and offer high-level feedback appropriate for a peer-  
33 reviewed, high-impact journal.

34 4. Deliver final recommendations (e.g., Accept, Minor Revision, Major Revision, Reject),  
35 each justified by the preceding review.

36

37

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4  
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22   lending help with the MiST test at their laboratory.

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