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(Article begins on next page)

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RILEM interlaboratory study on the mechanical properties of asphalt mixtures modified with polyethylene waste

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ABSTRACT

This research aims to determine if the observed improvements using polyethylene (PE) waste in asphalt binder translate into better performance at the asphalt mixture scale in the laboratory environment while overcoming the stability and homogeneity issues experienced at the binder level. This is accomplished through a round-robin multinational experimental program covering four continents, with the active participation of eleven laboratories within the RILEM TC 279-WMR. PE modified AC16 mixtures were prepared employing the dry process using local materials with the PE waste provided by one source. Various mechanical tests were performed to investigate the compactability, strength, moisture sensitivity, stiffness and permanent deformation. Compared to the control mixtures, the following observations were made for PE modified mixtures: easier to compact, lower time dependence of stiffness, higher elastic behavior, lower creep rate, and higher creep modulus. Furthermore, cyclic compression test results showed that the resistance to permanent deformation is improved when using PE in asphalt mixtures, whereas the wheel tracking tests showed relatively similar or better results when 1.5% PE was added to the control mixture. The wheel tracking test results in water showed an increase in deformation with increasing PE content. The interlaboratory investigation showed that the use of PE as a performance-

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enhancing additive in asphalt pavements is a viable, environmentally friendly option for recycling waste plastic and could potentially reduce the use of polymer additives in asphalt.

1. Introduction

Accumulation of waste is one of the significant challenges of this century. An essential step toward a zero-waste society is reducing and reusing waste. The asphalt community is taking important steps to make progress in this direction: on the one hand, reusing reclaimed asphalt and; on the other hand, using waste and marginal materials as a substitute for virgin materials. It was shown that using waste and marginal materials in roads has the potential to reduce CO_2, energy demand, and costs (Punith and Veeraragavan, 2007; Moghadas Nejad et al. 2014; Poulikakos et al., 2017; Lu and Giustozzi, 2022). At the same time, such alternative materials used in pavements could achieve a similar or even better performance as conventional materials (Piao et al., 2021). However, the data is mostly from laboratory tests as these alternative materials have a varied technology readiness level (TRL) worldwide. In the case of plastics, such as polyethylene (PE), the TRL level has reached 5–7, indicating that pilot projects have been implemented in the field in India, China, and Singapore, among others (Piao et al., 2021).

RILEM Technical Committee (TC) 279-WMR (Valorisation of Waste and Secondary Materials for Roads) investigates the use of waste and marginal materials in roads. Various technical groups (TG) deal with the use of alternative materials as binder additives (TG1 and TG2) or aggregate substitutes (TG3) and the life cycle assessment thereof (TG5). The influence of PE waste as a binder additive was investigated in TG1 (Tušar et al., 2022; Wang et al., 2022a, 2022b). Due to the promising results and the revealed challenges, this publication reports on the use of the same PE waste as a performance-enhancing additive in asphalt mixtures using the dry process.

Past research investigated the possibility of reusing different waste plastics (PE included) in asphalt mixtures using both the dry (i.e., direct addition to the mix) and the wet (i.e., bitumen modification) methods (Brasileiro et al., 2019; Lu and Giustozzi, 2022). Such studies seemed to demonstrate the feasibility of recycling waste plastics in asphalt mixtures while achieving similar or even enhanced performance in terms of permanent deformation and fatigue resistance as well as bearing capacity and durability compared to the corresponding reference mixtures (Moghadas Nejad et al., 2014, Lastra-Gonzalez et al., 2016; Giri et al., 2018; Giustozzi et al., 2022). Studies have reported that the addition of waste plastic in asphalt binders caused an issue with storage stability and segregation (Kakar et al., 2021; Naskar et al., 2010). Efforts have been made to improve this; for example, Wang (2022) studied the storage stability of PE-modified asphalt. It was shown that it is difficult to solve the compatibility problem between PE and base asphalt by adding a stabilizer. The experimental result showed that styrene-butadiene-styrene block copolymer (SBS) could be melted with waste PE with a twin-screw extruder to prepare PE-SBS modifier. The PE-SBS modified asphalt prepared was stable in storage. Nguyen Ann et al. (2022) showed enhanced compatibility between the components and increased mechanical performance of asphalt and polyethylene (PE) through the use of dispersed/exfoliated nanoclays (NC) that promote a greater adhesion between asphalt and polyethylene, thereby leading to significant enhancements in all mechanical properties. However, the need for prior blending of binder and plant adjustments can be avoided in the dry method, which is currently considered a more promising technique. Moreover, almost all the previous studies rely on the results measured in a single laboratory. In this present study, a round robin test was conducted to better understand the effect of waste PE on the mechanical properties of modified asphalt mixtures. This effort reported expected values and expected dispersion of results for the first time. Therefore, a detailed investigation of PE blended mixtures using the dry process is of high importance to evaluate the key performance characteristics.

2. Research objectives

The research results from TG1 of RILEM TC 279-WMR have shown that the use of PE waste can be a viable option to improve the mechanical properties of asphalt binders. However, several issues were also observed, likely due to an incomplete melting of the waste plastic into the asphalt binder, that led to heterogeneous test results and unstable samples, especially at high temperatures (Tušar et al., 2022; Wang et al., 2022a, 2022b).

The present research focuses on determining if the improvements observed at the binder scale translate into comparable or better performance at the laboratory asphalt mixture scale while overcoming the abovementioned issues. This is accomplished through a round-robin multinational experimental program with the active participation of eleven laboratories covering four continents. The laboratories consisted of three from Italy, Brazil, Serbia, Canada, India, Belgium, Switzerland, Finland, and Spain, investigating various aspects of asphalt concrete performance, including compactability, water sensitivity, stiffness, and resistance to permanent deformation, thereby gaining knowledge on the strengths and weaknesses of this important waste stream for use in asphalt. The interlaboratory investigation aims to take a critical look at using PE as a performance-enhancing additive in asphalt and to provide confidence and guidance for using this waste material in roads.

3. Materials and mixtures

A detailed mixture and sample preparation method was developed within the TG so that all laboratories followed the same protocol. The materials used in this research were aggregate and neat bitumen obtained from each participant's country, along with PE waste plastic distributed from a single source to all laboratories. These materials were used to prepare asphalt concrete with a nominal maximum aggregate size of 16 mm, which is typically used to construct a binder layer (AC16 bin).

The aggregates and filler used in this study were of limestone origin, with the exception of two laboratories. Lab 2 used granite aggregates with 1% hydrated lime added to improve the aggregate-bitumen bond, whereas Lab 6 employed natural river sand as part of the sand fraction.

A 50/70 penetration grade bitumen, collected by each laboratory separately, was used to produce asphalt mixtures. Only one laboratory (Lab 8) used a 70/100 penetration grade bitumen. The penetration values were between 60 and 78 dmm, and the softening point temperature values were between 42 and 54 °C.

The same PE waste plastic source already used in TG1 of TC 279-WMR for the modification of bitumen was obtained from a plastic recycling centre in Switzerland and samples were distributed to the laboratories. The waste plastic was sampled from three batches (denoted as PE Batch 1, PE Batch 2, and PE Batch 3, with the second digit indicating the received batch number; for example, batch 3-1 means the first delivery of batch 3). Each laboratory received around 1.4 kg–4 kg of material from a single batch. Before further application, each laboratory sieved the material through the 11.2 mm sieve, discarded the retained material (if any), and determined the particle size distribution of the remaining sample. Fig. 1 displays the gradation of waste plastic samples from multiple laboratories. It can be seen that there is a certain variation among the three batches, with batch 3 being coarser.

The suggested aggregate gradation bands and the grading curves of the individual laboratories are displayed in Fig. 2. A binder content of 4–6% and voids content of 4–6% was recommended following the

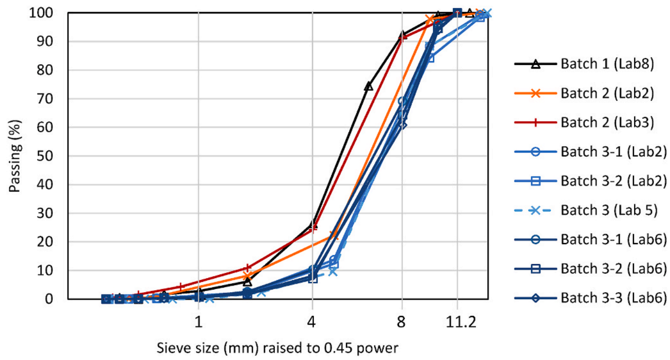

Fig. 1. Gradation of three PE batches reported by multiple laboratories.

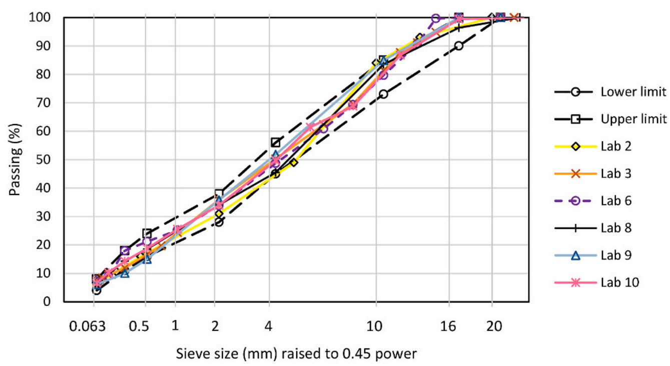

Fig. 2. Aggregate gradation of all participating laboratories and upper and lower limits.

Marshall compaction method (EN 12697-30, 2018) by applying 75 blows per each side or using a gyratory compactor (EN 12697-31, 2019) using 100 gyrations. Following the experience from the binder experiments where 5% PE per weight of binder was used, each laboratory prepared mixtures with the addition of at least one dose of PE waste plastic. All laboratories used 1.5% plastic waste by weight of asphalt mixture, while other amounts were optional. Results were reported for the following PE dose by mixture weight: 0, 0.25, 0.5, 1.0, 1.5 and 5%. Detailed information about the mixtures of individual laboratories is given in Table 1 including the amount of PE waste plastic applied and the resulting volumetric properties of asphalt mixtures with average values and standard deviations. Volumetric properties of all reference asphalt mixtures are graphically presented with triangular representation in Fig. 3. The lines indicate the boundaries of the requirements, with magenta for bitumen content in % by weight of aggregates (4%–6%) and red for void content (4%–6%). It is noteworthy that although all laboratories used local aggregates, the average aggregate density was 2.702 Mg/m^3 with a standard deviation of 0.098 Mg/m^3, indicating that the aggregates were similar in density regardless of geographical distribution.

4. Methods

4.1. Specimen preparation

The aggregate fractions and the bitumen were preheated in an oven at 160 °C for at least 3 h (EN 12697-35, 2016). The designated amount of PE waste plastic (Table 1) was kept at room temperature and then premixed with the hot aggregate by hand or in an automatic mixer for 1 min in the case of small batches (approx. 10 kg) or for a longer time for larger batches (e.g., 7 min for a batch of 25 kg with 1.5% plastic, as reported by Lab3). In practice, the premixing was adapted to visually obtain a homogeneous mix of aggregate and PE particles. This kind of waste plastic addition can be considered a dry process. Although the mix temperature was above the melting temperature of PE, it was clearly observed that the plastic only partially melted (Fig. 4); aggregate coverage was partial and some plastic particles were still visible. This issue was further investigated in a parallel study using environmental scanning electron microscopy on compacted mixture after cutting and polishing the surfaces (Kakar et al., 2022). The observations showed that the plastic particles did not completely melt and were present as inclusions within the mastic.

The defined binder content (Table 1) was poured onto the blend at 160 °C and mixed for an additional 2 min for small batches or for a longer time in the case of large batches (e.g., 5–6 min for a batch of 25 kg with 0.25% and 1.5% plastic, as reported by Lab3). Finally, the filler kept at room temperature was added to the blend and mixed for one additional minute for small batches or 3–4 min for larger batches (batches of 25 kg with 0.25% and 1.5% plastic). Asphalt mixtures were then compacted at 155 °C, and samples were prepared depending on the mix characterisation tests required, using the following compactors:

Table 1
Asphalt mixture properties.

PE %	lab1	lab2	lab3	lab4	lab5	lab6	lab7	lab8	lab9	lab10	lab11	avg	SD
	Void content (vol. % in asphalt sample)												
0	5.30	3.60	3.54	5.60	4.30	5.54	6.59	4.50	6.00	3.70	5.54	4.93	1.05
0.25	5.10	4.00	3.48					4.50	5.80			4.58	0.91
0.5											5.14	5.14	
1.0											5.05	5.05	
1.5	4.80	5.00	5.93	4.40	4.50	4.59	3.13	4.50	4.70	5.70		4.73	0.76
5					5.47				2.70			4.09	1.96
	Maximum densities (Mg/m^3)												
0	2.540	2.500	2.550	2.548	2.462	2.492	2.448	2.506	2.512	2.535	2.601	2.518	0.043
0.25	2.535	2.500	2.525					2.496	2.510			2.513	0.017
0.5											2.588	2.588	
1.0											2.576	2.576	
1.5	2.538	2.400	2.485	2.493	2.400	2.419	2.419	2.446	2.450	2.465		2.452	0.045
5					2.283				2.346			2.315	0.045
	Bitumen content (w. % by weight of stone aggregate)												
0	4.00	4.70	4.10	5.00	5.00	4.71	5.4	5.00	5.30	4.88	5.26	4.80	0.44
0.25	4.00	4.70	4.10					5.00	5.30			4.62	0.56
0.5											5.29	5.29	
1.0											5.32	5.32	
1.5	4.00	4.70	4.10	5.00	5.00	4.71	5.4	5.00	5.30	4.90		4.75	0.43
5					5.00				5.30			5.15	0.21
	Aggregate density (Mg/m^3)												
0	2.708	2.693	2.725	2.766	2.660	2.664	2.661	2.714	2.736	2.744	2.846	2.702	0.098

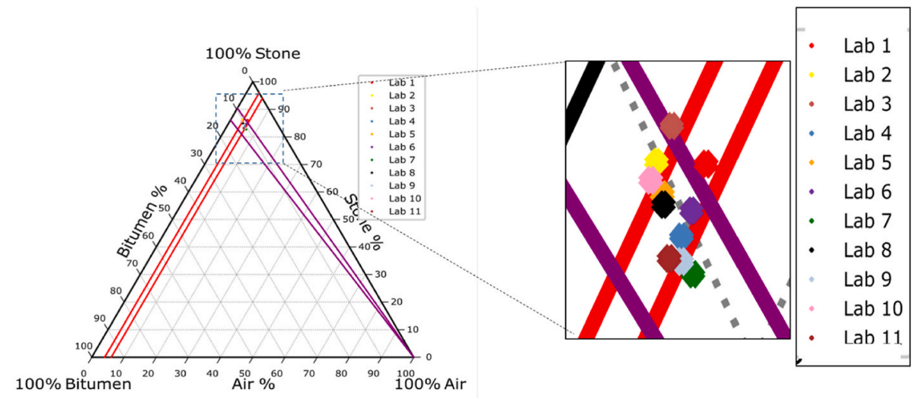

Fig. 3. Position of the volumetric properties of asphalt samples. Lines indicate boundaries of the requirements: magenta for bitumen content (% by weight of aggregates) 4%–6% and red for void content: 4%–6%.

Fig. 4. Preparation of asphalt mixture with PE waste plastic a) Hot aggregates and PE waste plastic; b) Addition of binder; c) Final mixture; d) PE waste batch 3; e) Partially melted plastic in the asphalt mixture (Courtesy Lab 6 and Lab 7).

Marshall (EN 12697-34, 2020), gyratory (EN 12697-31), and roller compactor (EN 12697-33, 2019).

4.2. Workability

The influence of PE addition on mixture workability was assessed through densification curves in the case of gyratory compaction. To this aim, the construction densification index (CDI) was calculated according to Bahia et al. (1998). This index measures the effort demanded to compact a given mixture, which is important to consider for implementing the modified mixtures in the field. The index takes the area of a compaction curve from the 8th gyration to a compaction degree of 92% of the maximum theoretical density of the mixture (Gmm).

For comparison, compactability was also determined according to European standard EN 12697-10, 2017. This European Standard applies to bituminous mixtures prepared in the laboratory or sampled from the plant and describes three test methods for characterizing the compactability of a bituminous mixture through the relation between its density or void content and the compaction energy applied to it, using an impact (Marshall) compactor, gyratory compactor, or a vibratory compactor. The results used were from the laboratories involved that used the gyratory compaction. In this case, the compactability K is defined as the slope of the regression line constructed by plotting the air voids versus the number of gyrations (log scale).

4.3. Tensile strength and moisture resistance

For each asphalt mixture, a set of cylindrical specimens was compacted by the laboratories to obtain the target air voids content given in Table 1. According to EN 12697-12, 2018, specimens were divided into two equally sized subsets, named "dry" and "wet". The dry subset was stored at room temperature (20–25 °C) while the other subset was saturated and conditioned in water at 40 °C for a period of 72 h. After conditioning, the indirect tensile strength (ITS) of each of the two subsets was determined (EN 12697-23, 2017).

ITS tests were conducted at 25 °C with a displacement rate of (50 ± 2) mm/min. ITS was calculated from the peak load diametrically applied to the cylindrical specimen using the following equation:

$$ITS = 2P/\pi dh \text{ [kPa]} \quad (1)$$

where P is the peak load [N]; d and h are the diameter and the height of the specimen [mm], respectively. At least three replicates were tested for each mixture type and average values were considered in the analysis.

The moisture sensitivity of asphalt mixtures was then determined in terms of ITS ratio (ITSR) as follows:

$$ITSR(\%) = \frac{ITS_{wet}}{ITS_{dry}} \times 100[\%] \tag{2}$$

where ITS_{wet} is the average ITS of the wet group [kPa] and ITS_{dry} is the average ITS of the dry group [kPa].

4.4. Stiffness

The stiffness modulus (*E*) was mostly evaluated using the pulse indirect tension test on cylindrical specimens (IT-CY), at 25 °C with 124 ms rise time and a maximum horizontal deformation of 9.0 μm (EN 12697-26, 2018 Annex C). Specimens were compacted through different methods, with Marshall or gyratory compactor, depending on the laboratory, with a diameter of 100 mm or 150 mm. Specimens have an average air void content of 4.5%, with values varying from 3.5% up to 5.9%, depending on the laboratory and the PE content. At least 10 conditioning pulses were applied vertically to allow the equipment to adjust the load magnitude and duration to obtain the necessary horizontal diametral deformation and rise time. Both measurements of the vertical and horizontal displacement allowed the calculation of the Poisson's ratio, which was used in the stiffness modulus calculation. In cases where determining the Poisson's ratio was unfeasible, a value of 0.35 was assumed, as recommended by the standard. The stiffness modulus was determined for each load pulse using the following equation:

$$E = \frac{F \cdot (v + 0.27)}{(z \cdot h)} \tag{3}$$

where *F* is the cyclic vertical applied load [N]; *v* is the Poisson's ratio [-]; *z* is the amplitude of the resilient horizontal deformation [mm]; and *h* is the specimen height [mm].

Stiffness measurements by Lab 4 were performed using the direct tension-compression test on cylindrical specimens (DTC-CY) (EN 12697-26 Annex D). Using a servo-hydraulic press system, the measurements were made in strain-control mode by targeting 50 μstrain to remain in the linear viscoelastic domain. Three 50 mm extensometers at 120° intervals around the specimen were used to measure axial strain. Measurements were completed at targeted temperatures of 25 °C and 35 °C at various frequencies (from 10 Hz to 0.01 Hz, for a total of seven frequencies). A thermal chamber was used for thermal conditioning; the temperature was monitored by three temperature probes glued to the specimen. Cylindrical samples were compacted using a Superpave gyratory compactor at a target voids content of 5%. Then, specimens were cored to obtain cylindrical specimens of 57 mm in diameter and 127 mm in height.

4.5. Resistance to permanent deformation

Two methods were used to investigate the resistance to permanent deformation at elevated temperatures; the cyclic compression test and the wheel tracking test.

4.5.1. Cyclic compression tests (CCT)

The European Standard EN 12697-25:2016 specifies three test methods for determining the resistance of bituminous mixtures to permanent deformation by means of cyclic compression tests, which ensure the ranking of various asphalt mixtures without a quantitative prediction of rutting in the field. This investigation was focused on a uniaxial cyclic compression test, where a cylindrical test specimen is subjected to cyclic axial stress with continuous measurement of permanent axial deformation. The cylindrical test specimen with a nominal diameter of 150 mm, conditioned at the testing temperature of 60 °C for at least 2 h before testing, was placed between two parallel loading platens. For the test method used in this study, the upper platen had a diameter of 100 mm, and no additional lateral confinement pressure was applied. A rectangular and periodical vertical stress pulse with a frequency of 0.5 Hz and pressure of 300 ± 2 kPa was applied to the specimen. The testing results are displayed as the cumulative axial strain of the test specimen, expressed in %, as a function of the number of loading cycles and denoted as creep curve. Generally, three stages can be identified on the creep curve. In stage 1, the initial part of the creep curve, the slope decreases with increasing loading cycles; in stage 2 (the middle part of the creep curve), the curve slope is quasi constant and can be expressed by the creep rate (f_c). Additionally, the exact turning point of the creep curve can typically be identified within this stage; in the last part of the curve (stage 3), the slope increases with an increase in loading cycles. At least two cylindrical specimens from every mixture were tested. The resistance to permanent deformation of asphalt mixtures is assessed by comparing accumulated axial strain, ε_n, creep rate in stage 2, f_c and creep modulus, E_n, calculated in accordance with the following equations:

$$\varepsilon_n = 100\left(\frac{u_n}{t_i}\right) \tag{4}$$

$$f_c = \frac{\varepsilon_{n_1} - \varepsilon_{n_2}}{n_1 - n_2} \tag{5}$$

$$E_n = \frac{\sigma}{10}\varepsilon_n \tag{6}$$

where;

ε_n is the cumulative axial strain of the test specimen after *n* loading cycles [%]; u_n, is the cumulative, permanent deformation of the test specimen after *n* loading cycles [mm]; t_i is the initial thickness of the test specimen [mm]; f_c is the creep rate [μm/m/loading cycles]; ε_{n1}; ε_{n2} are the cumulative axial strains of the test specimen after n_1, n_2 loading cycles [%]; En is the creep modulus after n loading cycles [MPa]; σ is the applied stress [in kPa].

4.5.2. Wheel tracking test

The wheel tracking test was conducted to determine the susceptibility of bituminous materials to deform under load at elevated temperatures. Two replicate asphalt mixture slabs with dimensions 500 mm (length) x 180 mm (width) x 50 mm (thickness) were prepared using a laboratory slab compactor. The test was conducted either in air or in water at 60 °C, as per EN 12697-22, 2021, Procedure B. According to this method, a pneumatic wheel loaded to 700 ± 10 N travels a total distance of 230 ± 10 mm at a frequency of 26.5 ± 1 cycles per minute while the resulting surface rut depth is measured after a selected number of passes.

To analyse the resistance of the asphalt materials to permanent deformation, the proportional rut depth (PRD) after 10 000 cycles and the wheel-tracking slope (WTS) were considered. WTS, in mm per 1000 load cycles, is calculated as follows:

$$WTS = \frac{d_{10000} - d_{5000}}{5} \tag{7}$$

where d_{5000} and d_{10000} are the vertical displacements after 5000 and 10 000 load cycles [mm].

5. Experimental results

5.1. Workability

The construction densification indices were calculated from several laboratories. While most of the participants compacted 150 mm samples with angles of gyration of 1.25°, a few laboratories conducted the compaction on 100 mm samples or with angles of gyration of 1.16 or 0.82°. The CDI values according to the PE addition percentages are shown in Fig. 5.

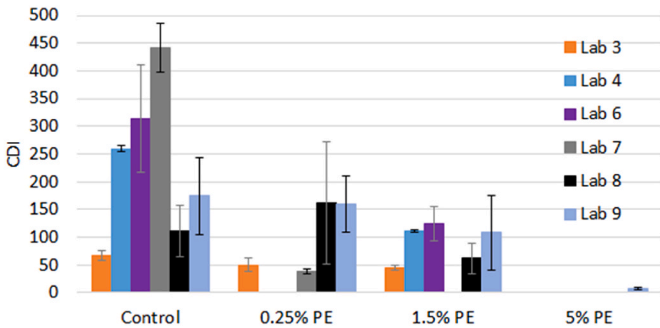

Fig. 5. Construction densification index (CDI) and its standard deviation for gyratory compacted samples (Lab 3 Angle = 1.16°; Lab 6 Angle = 0.82°; Lab 9 Diameter = 100 mm).

As shown in Fig. 5, the general tendency was for a decrease in the CDI with the addition of PE, indicating more ease in compaction. This tendency was the strongest with the results from Labs 4, 6, and 7, which had only one plastic content to compare with the control. The compaction from Labs 3 and 9 — which had two and three different plastic contents, respectively — also showed this tendency, although the reduction in CDI was not as severe as with the other labs. The CDI for the mixture with 5% PE was only 7.6 compared to 173.9 for the respective control. Only the results from Lab 8 did not show a clear reduction in CDI after adding PE, even if a lower average CDI can be noted for 1.5% PE mixture. It should also be noted that some CDI results had a very low standard deviation, while this was very high for other data sets. This does not seem to correlate with the angles of gyration.

Compactability was also calculated according to European standard EN 12697-10. Repeatability and reproducibility of compactability factors *K* (Fig. 6) were better than for CDI, leading to lower differences between laboratories. However, contrary to the CDI results, adding PE did not significantly improve the compactability. Furthermore, the *K* factor for samples with different contents of PE was very similar, leading to the conclusion that the PE content does not affect the compactability factor *K* significantly. It is noteworthy that the *K* factor fits the whole compaction curve from the first to the last gyration, whereas the CDI refers only to part of the gyrations, from the 8th gyration to a compaction degree of 92% of the maximum theoretical density of the mixture (Gmm). Comparing CDI and the EN method show that the CDI was more sensitive to the PE content.

5.2. Indirect tensile strength (ITS)

The effect of PE waste plastic on the mechanical properties of asphalt mixtures has been investigated by all laboratories involved through the ITS test, one of the most popular tests used for asphalt mixture characterization worldwide.

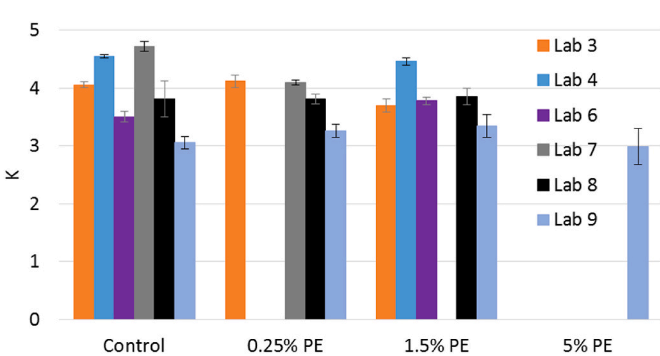

Fig. 6. Compactability factor K and its standard deviation for gyratory compacted mixtures.

To allow for direct comparison among laboratories, ITS results were normalized with respect to their PE-free control mixtures. Normalized ITS results in dry and wet conditions for each PE content are given in Figs. 7 and 8, respectively.

Fig. 7 shows that the ITS of mixtures containing PE is higher than that of the control mixtures. The only exceptions were Labs 1 and 10 for which a slight reduction in ITS was recorded. The increase in PE content showed a clear increasing trend in the ITS results of Labs 4,6,7,8,9. This was less evident but still confirmed by Labs 2, 3, 5 and 11.

Referring to the average values, for mixes with 0.25%, 0.5, 1.0, 1.5% and 5% PE waste plastic, normalized ITS in dry conditions increased by 5.6%, 8.9%, 7.46%, 28.3% and 50.5%, respectively. The absolute value of the results ranged from 1.09 MPa to 1.58 MPa with a standard deviation of 0.39 MPa–0.49 MPa.

The increase in the ITS can be attributed to the greater cohesion of the modified mastics provided by the dispersion of the PE waste plastic. This confirms previous studies by Capitão et al. (2022) that have reported that with the addition of PE plastic, the ITS increases compared to mixtures without PE plastic.

5.3. Water sensitivity

Normalized ITS results in wet condition for each PE content are given in Fig. 8. After conditioning in water, the trend remained unchanged, with the only exception being Lab 2, showing a reduction in ITS at 1.5% of PE content (Fig. 8). This could be attributed to the aggregate type being granite. Referring to the average values, for mixes with 0.25%, 0.5%, 1.0%, 1.5% and 5% PE waste plastic, normalized ITS in wet conditions increased by 4.5%, 2.4%, 1.27%, 24.7% and 36.9%, respectively. The absolute value of the results ranged from 0.98 MPa to 1.38 MPa with a standard deviation of 0.30 MPa–0.44 MPa.

Fig. 9 presents the variation in ITSR with PE content obtained from different laboratories. The average values at different dose of PE are also marked. The absolute value of the results ranged from 86.2% to 94.1%, with a standard deviation of 7.3%–14.5%. While the results from different laboratories are not in agreement with each other, a general trend of reduction in ITSR with the increase in PE content was observed, confirming the trend obtained by Ahmadinia et al. (2012), who used Polyethylene Terephthalate (PET) as an additive. Though it is anticipated that incorporation of PE in dry form will coat the aggregate surface and lead to resistance against moisture damage, the PE particles remain in a semi-solid form (and may not melt completely as discussed previously) and therefore act as an elastic filler. Moreover, it should be noted that the binder content for modified mixtures were similar to control mixtures in this study. This led to a change in density and air voids due to the interference of partially melted PE particles. These

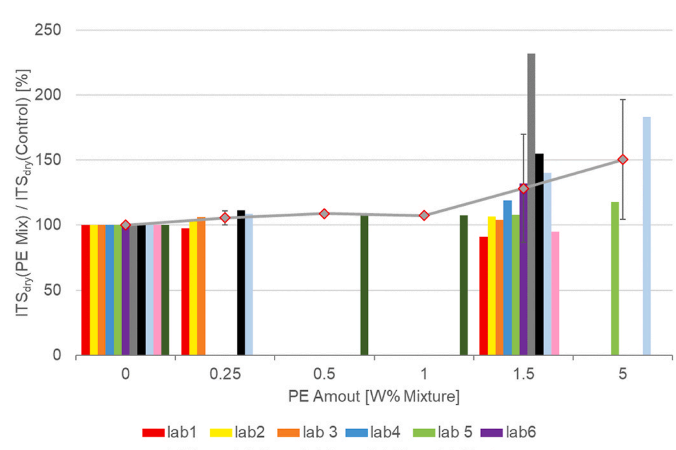

Fig. 7. Indirect tensile strength in dry state as a function of amount of PE normalized with respect to reference.

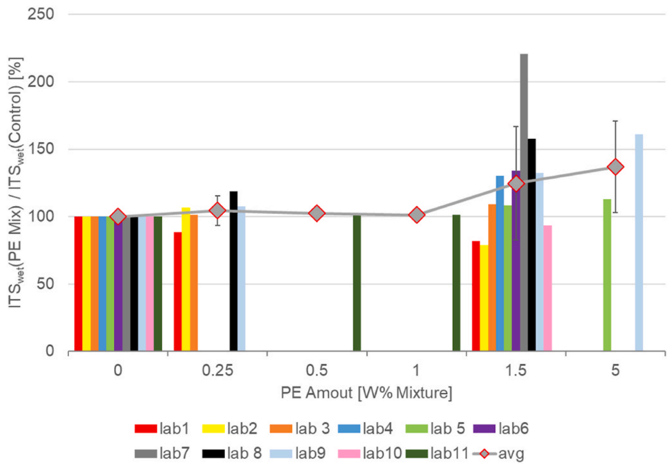

Fig. 8. Indirect tensile strength in wet state as a function of amount of PE normalized with respect to reference.

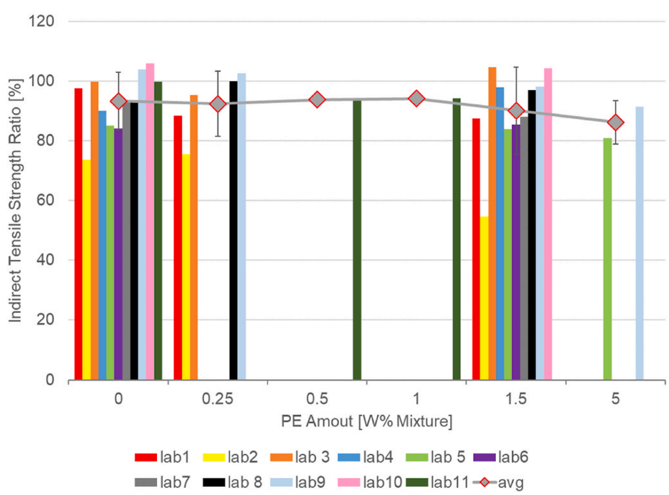

Fig. 9. Indirect tensile strength ration (ITSR) as a function of PE amount.

affect the propensity of the mixture to entrap different quantities of water, thus leading to a change in resistance against moisture damage. Lab 2 showed a remarkable reduction in ITSR value (from 74% for control mixture to 55% for 1.5% PE modified mixture) which is primarily attributed to the use of granite aggregates. Except for Lab 2, other labs reported ITSR values greater than 80%, irrespective of PE dose. Most of the highway agencies mandate a minimum ITSR of 80% or 70% as a check of moisture damage. From this perspective, most of the samples fulfilled the moisture sensitivity requirement.

5.4. Stiffness

Stiffness modulus of PE mixtures, obtained from 11 laboratories, were compared to the reference mixture (normalized) as a function of PE content. These percentage variations were represented by the bar chart, in Fig. 10, including a curve of the average obtained for all labs and the respective standard deviation. This approach allows to evaluate the effect of PE incorporation on the stiffness of the mixtures. In absolute terms for 0%, 0.25%, 1.5%, 5% PE content, an average stiffness modulus of 4865 MPa, 5484 MPa, 6780 MPa, 4782 MPa was measured respectively.

For most laboratories, with the exception of Labs 2, 4 and 10, with increasing PE content an increase in the stiffness modulus can be observed, as it has also been concluded by Lastra-González et al. (2022).

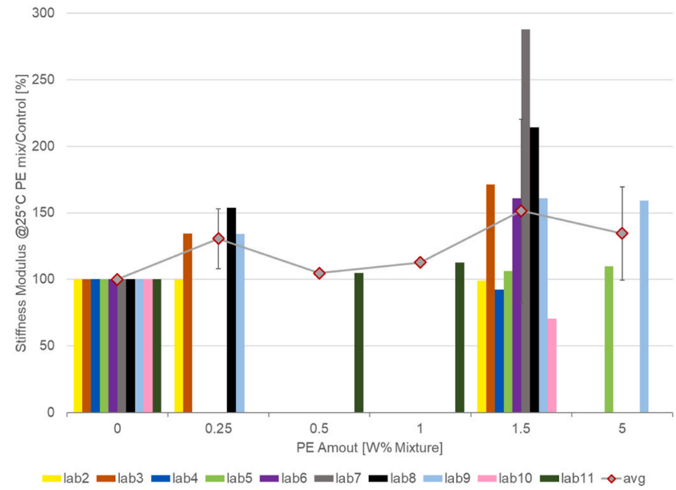

Fig. 10. Change in stiffness modulus as a function of PE amount normalized with respect to reference.

The increase was more noticeable for Lab 7, which reported an average stiffness modulus almost three times higher for the mixture with 1.5% PE when compared to the reference mixture. Lab 8 results also demonstrated higher sensitivity to the increase of PE amount, with gains of 53.9% and 114.3% for 0.25% and 1.5% PE contents, respectively, while Labs 3, 6, and 9 presented a percentual increase between 34.6% and 71.6%. Slight increases were also obtained for Labs 5 and 11, with minimum and maximum of 3.2% and 12.7%.

Only three labs (2, 4, and 10) presented results in which stiffness was reduced with the addition of PE. Lab 2 obtained a slight reduction in stiffness modulus, varying from 0.2% to 0.8%, for 0.25% and 1.5% PE mixtures, respectively. Lab 10 presented a decrease of 29.7% of the stiffness modulus when 1.5% PE was incorporated, varying from 1736 MPa to 1220 MPa. A possible reason for this reduction is related to the air voids content of the tested samples, which increased with the increase of PE, varying from 3.7% to 5.7%, respectively. While, in case of Lab 2 PE mixtures presented air void contents of 4.0% and 5.0% for 0.25% and 1.5% PE mixtures, respectively, and in comparison to the reference mixture with 3.6% air voids. Since Lab 4 applied sinusoidal continuous loading and not pulse with rest periods, these results will be discussed later. It is important to keep in mind that gradation, aggregate properties, volumetric properties, compaction energy, among other variables, can change the stiffness modulus of the materials. Still, based on the exploratory data analysis, it is possible to infer stiffer mixtures are obtained for higher contents up to 5% of PE.

As previously mentioned, one lab performed complex modulus measurements through axial sinusoidal load test on cylindrical specimens. The stiffness (i.e., the norm of complex modulus, $|E^*|$) and phase angle results obtained at 25 °C and 35 °C according to the testing frequency were plotted in Fig. 11. Red markers represent the plastic mixture results, and black markers the reference mixture results. As the objective was to evaluate the effect of plastic on mixture stiffness, the results of the asphalt mixture with 1.5% PE were compared with those of the reference mixture. The difference in stiffness values between both mixtures is plotted next to each testing frequency result.

An interesting observation could be made following the completion of the experimental test. According to the results, it seems that the PE modified mixture stiffness values are higher for some testing frequencies than the reference mixture. This is true for testing frequencies from 0.01 Hz to 1 Hz, where the stiffness difference ranges from 76 to 712 MPa. Conversely, the opposite trend can be observed for testing frequencies of 3 Hz and 10 Hz. The plastic mixture stiffness is lower than the reference mixture, with the difference ranging from −63 MPa to −638 MPa. Thus, a lower time dependence of the stiffness of PE modified asphalt mixtures

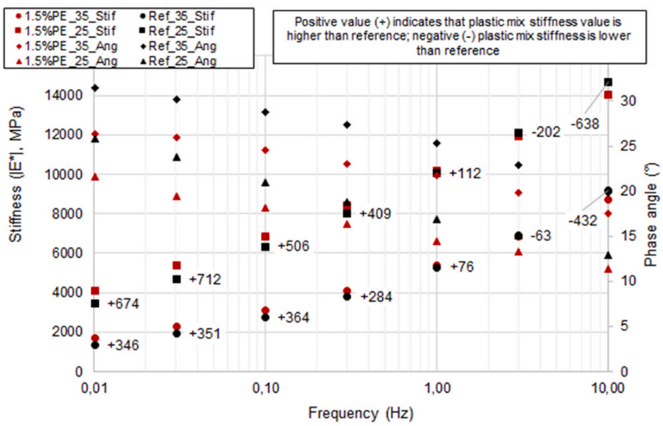

Fig. 11. Stiffness (Stif) and phase angle (Ang) as a function of frequency.

can be ascertained.

Regarding the phase angle, the results show that for both testing temperatures, the phase angle values of the PE modified mixture are lower than those for the reference mixture. Therefore, the PE modified mixture leans towards a higher elastic behavior than the reference mixture even if the difference between PE and reference mixture decreases when test frequency increases.

5.5. Permanent deformation

5.5.1. Cyclic compression test

Four laboratories performed the CCT tests showing, in some cases, that the cumulative axial strain of samples exceeded 4%, thus attaining failure (stage 3 of the curve appeared). The example shown in Fig. 12 indicates that the addition of PE reduced the cumulative axial strain considerably. Furthermore, Fig. 13 and Fig. 14 further confirm that the creep rate, fc, as well as the creep modulus, E_n, were significantly improved with the addition of plastic waste. This is a direct result of the amount of PE added, as demonstrated by the results from lab 5 and lab 8. Testing results presented here clearly indicate that the performance is positively affected by PE shreds content. Recall that the binder used by most laboratories was a 50/70 pen where according to the European standards should have a softening point between 46 °C and 54 °C. One lab (Lab 8) used 70/100 bitumen with softening point that should be between 43 °C and 51 °C (EN 12591, 2009). It should be noted that the melting temperature of PE is ca 120 °C (Kakar et al., 2021), so at the testing temperature binder starts to soften, while PE remains elastic, contributing to the low accumulated strain in the creep test.

5.5.2. Wheel tracking test

Out of the four laboratories that performed the wheel tracking test,

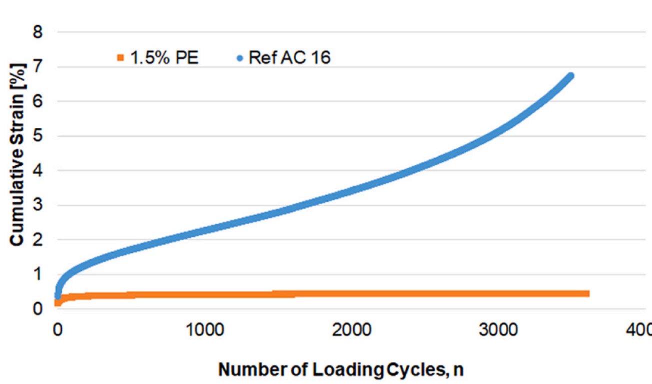

Fig. 12. An example of cumulative strain vs. number of loading cycles for two samples (courtesy lab7).

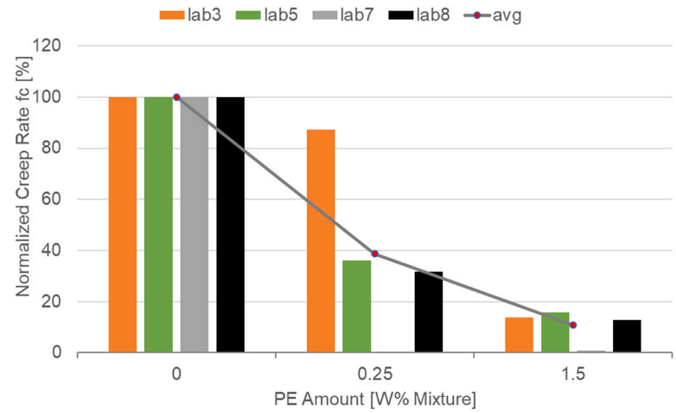

Fig. 13. Normalized creep rate f_c as a function of amount of PE normalized with respect to reference.

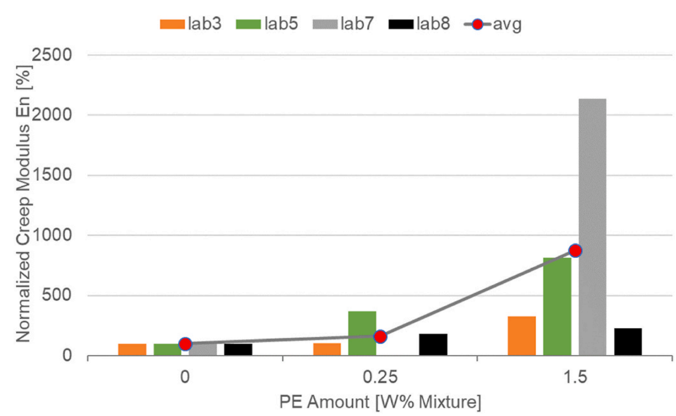

Fig. 14. Normalized Creep Modulus as a function of amount of PE normalized with respect to reference.

Lab 1 evaluated load deformation in water and the three other labs conducted tests in air. The results in Fig. 15 clear indicate an increase in proportional rut depth with increasing PE content for Lab 1 (tests in water), while the opposite trend, similar to those obtained by Lu and Giustozzi (2022) and Lastra-González et al. (2022), was found for Lab 6 (tests in air). Lab 2 reported a maximum rut depth for a PE content of 0.25%, whereas relatively similar values were obtained for the control and the mixture with 1.5% PE. Likewise, Lab 4 reported similar results for the control and the 0.25% PE mixes.

The wheel tracking slope results depicted in Fig. 16 tend to agree

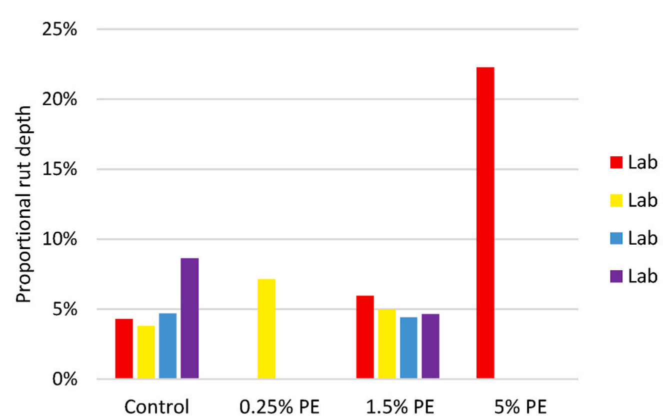

Fig. 15. Proportional rut depth from wheel tracking tests as a function of PE content.

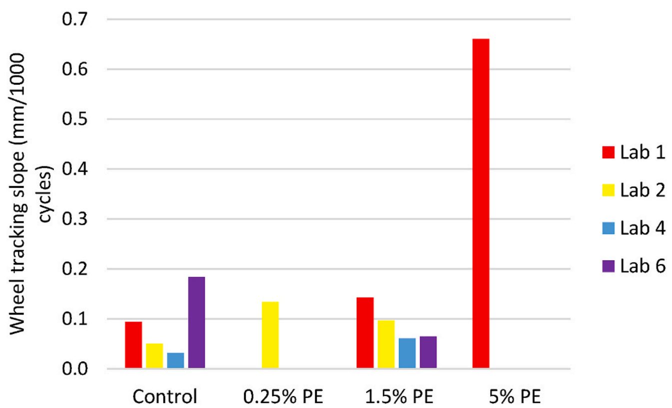

Fig. 16. Wheel tracking slope from wheel tracking tests as a function of PE content.

with the proportional rut depth: Lab 1 reported an increase with increasing PE content whereas Lab 6 found a decrease. The main difference in this case is that both Lab 2 and Lab 4 observed a greater WTS value for the 1.5% PE mix than for the control mix.

The overall results of the WWT tests indicate that caution should be exercised with the amount of PE the results in this interlaboratory study indicate that up to 1.5% of PE no adverse effects were observed; however, larger amounts can result in high rut depths.

6. Summary and conclusions

In the present research effort, an interlaboratory investigation was performed by TG1 and TG3 of RILEM TC 279-WMR to evaluate if the benefits observed by incorporating PE waste in asphalt binder translate into better performance of asphalt mixture while overcoming the stability and homogeneities issues experienced at binder level. Participants followed a developed protocol, used locally available bitumen and aggregates, while the plastic material was provided from a single source, although in different batches. A reference mixture and a corresponding PE modified mixture with different PE contents were prepared for the experimental campaign, consisting of a comprehensive series of tests, including strength (ITS and ITSR), stiffness (IT-CY and DTC-CY), and permanent deformation (CCT and wheel tracking). Based on the analysis of the laboratory measurements, it can be concluded that:

- Although the asphalt mixing temperature is above the melting temperature of PE, PE does not melt completely during the laboratory fabrication of asphalt concrete and acts as an elastic body embedded in the mixture;
- The addition of PE led to a lower Construction Densification Index (CDI), which indicates easier compaction; Comparing the CDI and the EN methods show that the CDI was more sensitive to the PE content and could distinguish between different mixtures;
- The addition of PE led to a higher Indirect Tensile strength (ITS), which may be attributed to the higher cohesion in the mastic phases caused by the dispersion of PE waste plastic. An overall and slight increase in ITS was observed with the PE content.
- PE modified mixtures (PE content less than 1.5%) were not more sensitive to water in comparison to reference mixture. One exception was the data from the lab that used granite aggregates that are known to be water sensitive;
- Stiffness of the PE modified mixtures tends to increase for higher percentages of PE (up to 5.0%), based on the pulse indirect tension test;
- Under axial sinusoidal load, higher stiffness is observed for lower testing frequencies. It is possible to ascertain that 1.5% PE modified mixture is less time dependent compared to the reference mixture;
- Regarding viscoelastic linear response, PE modified mixture demonstrates higher elastic behavior than the reference mixture;
- PE modified mixtures showed lower creep rate and higher creep modulus in comparison to the reference mixtures;
- The resistance to permanent deformation is improved when using PE in asphalt according to the cyclic compression test results. The results from wheel tracking tests in the air showed relatively similar or better results when 1.5% PE was added to the control mix. The wheel tracking test results in water showed increased deformation with increasing PE content. At higher PE dose (>5%) caution should be exercised as rutting can be a problem.
- Based on the results investigated here, the optimal PE content is 1.5% of mixture mass.

The fatigue properties, low temperature cracking resistance, and crack propagation will be investigated in future studies. Although this study did not use any modifiers, various efforts are underway to enhance the compatibility of PE with the base asphalt, such as PE-SBS modified asphalt (Wang, 2022) or dispersed/exfoliated nanoclays (NC) and polyethylene (PE) in asphalt (Nguyen Ann et al., 2022) leading to significant enhancements in all mechanical properties. More efforts in this direction are recommended. As with any waste or marginal material used in asphalt mixtures, the environmental susceptibility of such additives should be assessed, such as leaching and microplastics, as well as a life cycle analysis. Furthermore, a life cycle cost analysis would be beneficial in assessing the economic viability of using such products as the wider uptake of this technology will be limited without economic benefits. A previous study (Piao et al., 2021) showed that the technology readiness level (TRL) for using alternative materials for roads vary greatly worldwide. This implies that there is lack of widespread know-how which leads to lack of performance or trust in the product. Therefore, informing the stakeholders of the advantages and providing incentives and standards would help the implementation of using alternative materials for roads. This interlaboratory investigation aimed to take a critical look at using PE as a performance-enhancing additive in asphalt and to provide confidence and guidance for using this waste material in roads.

CRediT authorship contribution statement

Lily D. Poulikakos: Conceptualization, Resources, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Writing – original draft, Writing – review & editing. **Emiliano Pasquini:** Conceptualization, Resources, Data curation, Formal analysis, Methodology, Project administration, Writing – review & editing. **Marjan Tusar:** Conceptualization, Resources, Formal analysis, Investigation, Methodology, Project administration, Writing – review & editing. **David Hernando:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Di Wang:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Peter Mikhailenko:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Marco Pasetto:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Andrea Baliello:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Augusto Cannone Falchetto:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Miomir Miljković:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Marko Orešković:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Nunzio Viscione:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Nikhil Saboo:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Gabriel Orozco:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Éric Lachance-Tremblay:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Michel**

Vaillancourt: Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Muhammad Rafiq Kakar:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Nicolas Bueche:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Jan Stoop:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Lacy Wouters:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Davide Dalmazzo:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Gustavo Pinheiro:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Kamilla Vasconcelos:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing. **Fernando Moreno Navarro:** Data curation, Formal analysis, Investigation, Methodology, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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