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Article

A Comparative Study of the Effect of Moisture Susceptibility on Polyethylene Terephthalate–Modified Asphalt Mixes under Different Regulatory Procedures

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Abstract: Water damage is one of the main causes of road deterioration during its lifespan, leading to a decrease in the structural and functional qualities of the road surface. Moreover, the management and disposal of polyethylene terephthalate (PET) bottles at the end of their lifecycle are becoming increasingly complex challenges. Hence, this study investigates the feasibility of incorporating crushed PET bottles into the production of asphalt mixtures, considering different PET quantities (6%, 10%, 14%, 18%, and 22%) and two incorporation processes in the mixture design (dry process and modified dry process). PET-modified mixtures' volumetric properties, Marshall parameters, and moisture susceptibility characteristics were evaluated and compared with PET-free asphalt mixtures. The results indicated that PET content significantly influences the properties being assessed, and the modified dry process yields a higher resistance to moisture susceptibility. Finally, the obtained TSR (tensile strength ratio) results based on European standards are compared with those obtained using American standards, in an aim to comprehend and assess the testing methods, result reliability, and applicability.

Keywords: asphalt mixes; moisture damage; indirect tensile strength; polyethylene terephthalate; international standards

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1. Introduction

Asphalt mixtures are widely employed in road construction, and they play a key role in highway infrastructure by offering a durable and traffic-resistant surface [1]. However, as the volume and intensity of traffic increase, as well as the effects of climate change, innovative approaches need to be developed to improve the quality and performance of these mixtures [2].

One of the critical challenges in the road construction industry is to reduce environmental impacts and promote more sustainable practices [3]. In 2016, the European Commission published the report"EU Green Public Procurement Criteria for Road Design, Construction and Maintenance" [4], which addresses several environmental issues related to road infrastructure construction. This report presents a set of environmental criteria by which the most complex processes related to road construction works are assessed [5]. Technological advances, widespread industrialisation and consumer habits have intensified the accumulation of plastic waste, and the recycling and reuse of discarded materials have become areas of research and development of great interest [6]. In this context, through the Paris climate agreement, countries have committed to improving plastic design and isolating their production from fossil resources, reducing greenhouse gas emissions [7].

From a sustainability perspective, environmental impacts, economic benefits and pavement performance must be critically considered. The combination of waste materials in asphalt mixes addresses three environmental issues: solid waste management, air pollution and global warming [8].

One of the most essential industrial inventions has been plastic. It is a material with excellent properties, high durability and strength for its weight, easy handling and cheap production [9,10]. Global plastic production has grown significantly in recent years and is expected to exceed 35,000 billion metric tons by 2050 [11]. Among its main applications worldwide, the packaging industry (41.9%), construction (22.8%), the automation industry (11.2%) and electronics (7.3%) stand out [12].

Polyethylene terephthalate (PET) is a semi-crystalline polymer with good mechanical properties and is considered a thermoplastic polyester [13]. Since 1980, one of its most important applications has focused on producing beverage bottles, reaching a consumption in 2014 of 41.56 Mt, with an expected growth of 73% by 2025 [14]. Currently, the world's largest manufacturer is China (27%), followed by Europe (17%), North America and Canada (17%), and South America (7%) [15].

There are numerous studies on the use of recycled PET in asphalt mixes. In 2018, García-Travé et al. published a survey of the mechanical performance of SMA (stone mastic asphalt) mixes made with binders modified with re-covered geomembrane as an additive to improve the properties of SMA mixes used in pavements. Geomembranes previously used in landfill waterproofing projects were collected and processed to obtain a fine powder incorporated into bituminous binders. The mechanical properties of the mixes, such as compressive strength, tensile strength, were analysed, and fatigue resistance of the SMA mixes modified with the recovered geomembrane. The results showed that the addition of recovered geomembrane in the bituminous binders had a positive effect on the mechanical performance of the SMA mixes, improving the compressive strength, tensile strength and fatigue resistance of the modified mixtures compared to the reference mixtures without additive. A better binder–aggregate interaction was observed due to the presence of the recovered geomembrane, which increased the strength and durability of the mixes [16].

Due to the high resource consumption involved in road construction, researchers are focusing on developing more sustainable pavements using alternative materials, such as plastic waste, as a substitute for aggregates in asphalt mixes. Rivera et al. developed a study on the environmental impact assessment of plastics and polymer recycling from virgin use to post-consumer recycling as fibre or composites in the paving industry, applying the life cycle assessment (LCA) methodology. The study concluded that the use of PET in asphalt mixes results in a decrease of up to 38% in the overall environmental savings in terms of environmental score in favour of sustainable pavement alternatives [17].

In 2021, Tauste-Martínez et al. published a study on the effect of recycled polymers on the long-term performance of bituminous materials used in pavements. For this purpose, a multi-scale evaluation was carried out to analyse how recycled polymers influence the properties and performance of bituminous materials. Different recycled polymers from plastic waste were collected, characterised, and incorporated into bituminous mixtures in different proportions. Laboratory tests were carried out to evaluate the mechanical and rheological properties of the mixtures modified with recycled polymers. Long-term performance evaluation was done using numerical modelling and finite element simulation. The behaviour of the mixtures was analysed over time, and aspects such as permanent deformation, fatigue and cracking resistance were evaluated. The study results showed that adding recycled polymers to bituminous mixtures positively affects several critical properties of the materials. Improvements were observed in permanent deformation resistance, load-carrying capacity, fatigue resistance and cracking resistance. In addition, numerical modelling provided a better understanding of the mechanisms occurring at the microstructural level and their influence on the long-term behaviour of materials modified with recycled polymers [18].

According to Nisma Agha et al., PET-modified asphalt mixtures are an economical solution for road construction and maintenance and have significant advantages in terms of sustainability. This study evaluated the performance of polyethylene terephthalate (PET) modified hot mix asphalt using 2%, 4%, 6%, 8% and 10% PET. Following the determination of the optimum bitumen content, samples of modified HMA were prepared and tested using dry and wet mixing techniques, including moisture susceptibility testing (ALDOT-361-88), indirect tensile fatigue testing (ITFT-EN12697-24) and Marshall stability and flow tests (AASHTO T245-90). The dry mixing technique showed better resistance against fatigue cracking, stability and flowability. In comparison, wet mixing gave better results regarding resistance against moisture damage, the optimum PET content being 6% [19].

Figure 1 shows the research flow of this work. This article aims to analyse the effects of incorporating polyethylene terephthalate in bituminous mixtures and to evaluate its influence on water resistance based on three methods established by different standards (UNE, ASTM and AASHTO). The use of these standards and not others (e.g., the Texas boiling method) is based on their relevance to the road industry, their international recognition and acceptance, their more accurate and controlled approach, and regulatory and contractual compliance. The experimental study includes different percentages of PET in asphalt mixes, comparing the results to conventional reference mixtures. The results presented here are expected to contribute to advancing more sustainable practices in road construction, fostering the circular economy and improving the quality and performance of road infrastructure.

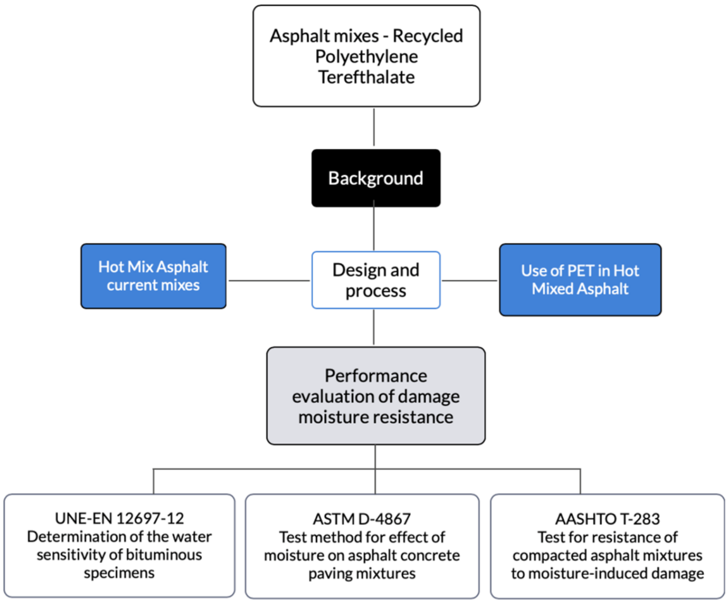


Figure 1. Research flow.

2. Materials and Methods

2.1. Aggregates

The aggregate used in the investigation is porphyritic igneous with a nominal maximum size of 16 mm as a coarse aggregate and fine aggregate fraction. The physical characteristics and the limits of the Spanish standard for the maximum traffic level are given in Table 1.

Table 1. Characterisation of the natural aggregates used in the investigation.

Aggregate	Test	Value	Limits
Coarse	Specific weight (g/cm^3)	2.796	-
	Slab index (%)	<1%	≤20%
	Los Angeles abrasion (%)	13	≤15%
	Water absorption (%)	0.60	<1%
Fine	Specific weight (g/cm^3)	2.726	-
	Sand equivalent	77	>55

Figure 2 presents the particle size distribution used in this study, commonly applied as a surface layer in Spain.

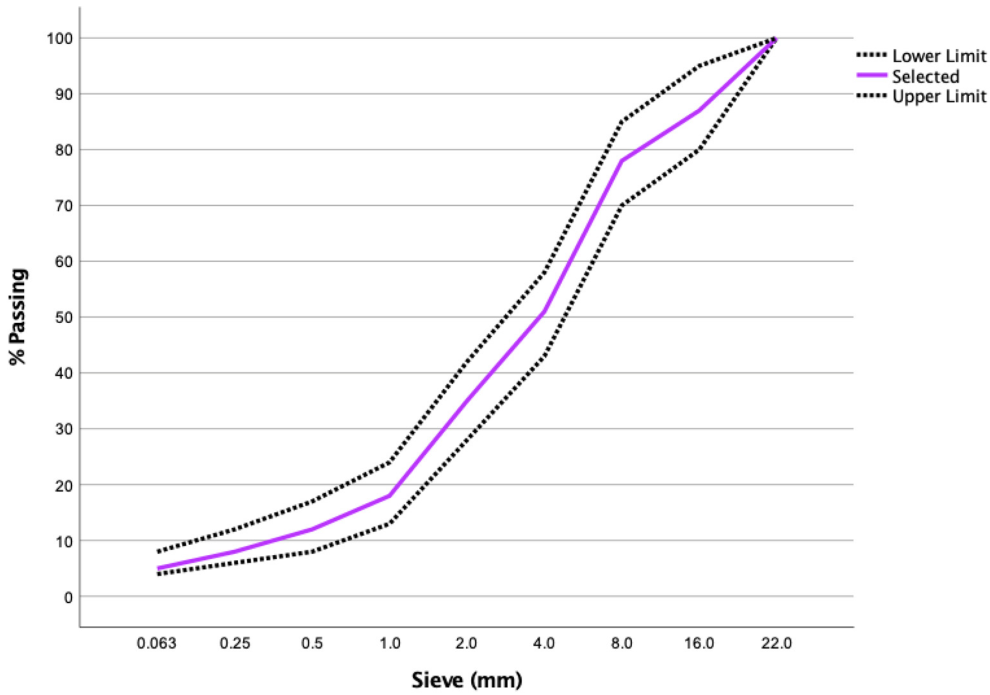


Figure 2. Aggregate particle size distribution.

2.2. Bitumen

The bitumen used is a CA-24 asphalt cement [12]. The essential specifications of the material are shown in Table 2.

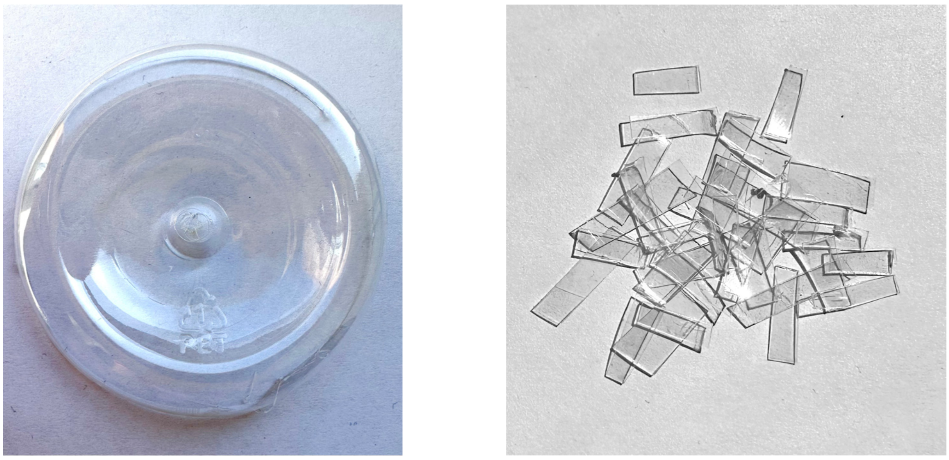
Table 2. Main physical characteristics of CA-24 asphalt binder.

Properties	Value	Limits
Penetration at 25 °C (0.1 mm)	54	50–70
Softening point (°C)	50	46–54
Frass breaking point (°C)	−13	≤−8
Specific weight (g/cm^3)	1.033	-

2.3. Polyethylene Terephthalate (PET)

Polyethylene terephthalate, also known as PET, is a transparent polymer with good mechanical properties and dimensional stability under a variable load that can improve the mechanical properties of the asphalt mixture. Based on the physical properties of the polymer, it reaches a glass transition temperature of 70 °C, i.e., it changes its internal composition, modifying its texture, and providing good stiffness and permanent deformation characteristics. The collected waste PET bottles were washed, dried and cut with scissors into small pieces, as shown in Figure 2. The length of the PET fibres is 10 mm, and the specific weight obtained is 0.90 g/cm^3.

A detailed illustration of the appearance of this material can be observed in Figure 3.



(**a**) (**b**)

Figure 3. Polyethylene Terephthalate (PET) used in the investigation: (**a**) Image of the beverage bottle; (**b**) 10 mm long cut PET fibres.

2.4. Dosage, Production and Air Void Content

The bituminous mixtures were dosed by European standards, following the established procedures, criteria, and related parts. Dosing refers to determining the appropriate proportions of aggregates, binders and additives to obtain a bituminous mixture with the required mechanical and performance properties. To this end, tests are carried out to determine the properties of the materials used in the mixture, as indicated in the previous point. Subsequently, the appropriate type of bituminous mixture is determined, considering factors such as expected traffic, climatic conditions and the characteristics of the existing pavement. In this case, a semi-dense AC16 S mixture with a maximum aggregate size of 16 mm is considered for the wearing course with a minimum binder content of 4.5%. The Marshall method was used for the design, whereby the appropriate proportions of aggregates, bitumen and PET were calculated to achieve the desired mechanical properties.

Table 3 summarises the number of samples manufactured according to the different percentages of PET and the procedures used (according to European and American standards) to manufacture each sample.

Table 3. Summary of the number of test pieces produced as a function of % PET.

Asphalt Mixture	% PET	N° Samples	Normative
M_1	0 (ref.)	16	European Standard
M_2	6	16	
M_3	10	16	
M_4	14	16	
M_5	18	16	
M_6	22	16	
M_7	0 (ref.)	12	ASTM Standard
M_8	10	12	
M_9	0 (ref.)	12	AASHTO Standard
M_{10}	10	12	

For the research, 144 samples were manufactured using the procedure established by determining the water sensitivity of bituminous specimens, where the first eight samples are called reference mixtures. The remaining 40 correspond to samples manufactured with different percentages of PET (6%, 10%, 14%, 18%, and 22%).

In addition, 48 samples were manufactured according to American standards using the procedure established for the determination of the effect of moisture on asphalt concrete paving mixtures and the method of test for resistance of compacted asphalt mixtures to moisture-induced damage. A total of six samples was manufactured for each test (six samples for the reference mixtures and another six with 10% PET), as the results obtained with this additive content exceeded the minimum TSR values established by European standards.

The manufacturing process was carried out in accordance with UNE standards. The aggregates were previously heated in a laboratory oven at 180 °C and the bitumen at 155 °C. Once the materials were heated, the aggregate was homogeneously mixed with the PET particles (according to the quantity per series) for 2 min to distribute the polymer without melting it. The bitumen and mineral filler were added and mixed until a homogeneous mixture was obtained. It should be noted that the incorporation of the polymer corresponds to a dry process, where it was mixed with the aggregate before the binder was added. This procedure was chosen because the melting temperature of PET is higher than the manufacturing temperature of the Marshall test samples (150–170 °C). For the compaction process, 50 blows per side were applied with an automatic Marshall hammer, which was necessary to increase the percentage of voids in the sample intentionally. Once compacted, the sample was demoulded at 20 ± 5 °C. In addition, a second mixing process called "modified dry" was carried out whereby the polymer was mixed with the binder before being added to the aggregate for further mixing.

It is important to note that the American standard indicates a different procedure from the European standard. After mixing, the sample must be left to stand in an oven at 60 °C for 16 h before compaction, which is considered "short-term ageing". Subsequently, the temperature is increased ad hoc to place it in the moulds and proceed with the compaction. Another important consideration is that no specific number of blows per face is required. However, ranges of void percentages are considered, i.e., for the ASTM standard, air void values between 6% and 8%, and for the AASHTO standard, between 6.5% and 7.5%, are indicated.

2.5. Indirect Tensile Strength on Moisture Damage

The accumulated damage in the different layers of pavement construction is the leading cause of failure in asphalt pavements [20]. This can be due to water seepage into the asphalt pavement structure, which modifies the mechanical properties such as strength, stiffness and durability [21]. The adhesiveness of an asphalt mixture is determined from the bond of the aggregate–binder system, and water damage can occur under two conditions: (a) when the asphalt binder loses adhesive properties, or cohesion occurs; (b) when the aggregate–binder adhesiveness is reduced. Both conditions result in stripping, which weakens the adhesion between the asphalt binder and the aggregate particles, thus affecting the chemical composition and their bond [22]. Therefore, the specific properties of these materials, as well as the properties of the mixture, significantly impact the performance of the asphalt pavement [23].

There are aggregate parameters such as surface texture, porosity, absorption, cleanliness and energy, which together with the binder chemistry and the bond of the binder-aggregate system affect moisture susceptibility [24]. In addition, PET with high surface energy can increase the surface energy of the modified binder, improving water resistance. A summary of the moisture behaviour of the asphalt mixture using PET waste as an additive is shown in Table 4.

Table 4. Moisture performance of asphalt mixes modified with PET waste.

Author/Year	Main Findings
Rui, L., et al. (2022) [25]	"Innovative application of waste polyethylene terephthalate (PET) derived additive as an antistripping agent for asphalt mixture: Experimental investigation and molecular dynamics simulation". The results of experimental tests and simulations show that the PET-derived additive significantly improves the loosening resistance in asphalt mixtures. The additive helps reduce water adhesion to aggregates and improves the bonding capacity between aggregates and the asphalt. This leads to a higher mixture stability and increased resistance to loosening.
Oldham, D., et al. (2021) [26]	"Reducing susceptibility to moisture damage in asphalt pavements using polyethylene terephthalate and sodium montmorillonite". The results show that adding PET and sodium montmorillonite clay improves the moisture damage resistance of asphalt pavements. These additives help to reduce water absorption by the binder and improve the cohesion and strength of the mixture. In addition, an improvement in the tensile strength and wear resistance of the modified pavements is observed.
Yengejeh, A., et al. (2020) [27]	"Reducing production temperature of asphalt rubber mixtures using recycled polyethylene wax and their performance against rutting". The results obtained indicate that the use of recycled polyethylene waxes allows a reduction in the production temperature of asphalt rubber mixtures. Furthermore, it can be observed that mixtures modified with these waxes show a better performance against rutting compared to mixtures without additives or with conventional additives.
Taherkhani, H., et al. (2019) [28]	"Investigating the mechanical properties of asphalt concrete containing waste polyethylene terephthalate". The results show that adding recycled PET to asphalt concrete improves some of its mechanical properties. An increase in tensile strength and higher fatigue strength were observed in the samples containing PET. This indicates that recycled PET can improve an asphalt pavement's durability and service life. In addition, the effect of recycled PET particle size on the mechanical properties of asphalt is highlighted, with smaller PET particles having a more significant impact on durability.
Lugeiyamu, L., et al. (2021) [29]	"Utilization of waste polyethylene terephthalate (PET) as partial replacement for bitumen in stone mastic asphalt". The results show that the addition of PET waste to stone mastic asphalt (SMA) as a partial replacement for bitumen positively affected the mechanical and performance properties of the mixture. An improvement in tensile strength, wear resistance and resistance to permanent deformation was observed in mixtures containing recycled PET. In addition, the effect of temperature on the properties of SMA with PET was analysed. It was found that the PET blends showed better performance at higher temperatures, indicating higher resistance to softening and deformation under hot climatic conditions.

The objective of the indirect tensile moisture damage test is to analyse whether the aggregate–binder system that makes up the bituminous mix is susceptible to the effect of water. Research has shown that the temperature must be concentrated between 10 and 25 °C for the resistance value to vary linearly. When working with temperatures above 30 °C, the values show dispersion, and the function becomes parabolic (viscous component).

Table 5 shows the parameters to be considered in the indirect tensile water sensitivity test for each procedure according to the UNE, ASTM, and AASHTO standards.

Table 5. Variables to consider in the test for the UNE, ASTM, and AASHTO standards.

Test Parameters	UNE-EN 12697-12	ASTM D4867	AASHTO T-283
Pre-ageing	Not required	Not required	Resting for 2 h at 25 °C. Later, in the oven for 16 h.
% Air voids	No limit (just use 50 blows per side of the sample)	6–8	6.5–7.5
Saturation	Do not expand sample by more than 2% of its initial volume.	55–80% 5 min at 525 mm Hg	70–80% for 5–10 min between 254–600 mm Hg
Ice and thaw cycle	Not required	Optional	−18 °C for 16 h.
Temperature and conditioning time	20 ± 5 °C for 16–24 h	60 °C for 24 h	60 °C for 24 ± 1 h
Test temperature ITS	15 ± 5 °C at 51 mm/min	25 ± 1 °C at 51 mm/min	25 ± 1 °C at 51 mm/min
TSR (%)	85	75	80
Visual analysis	Not required	Not required	Only requires doing so

Finally, the maximum stress is determined, calculated as a function of the compressive load applied along the diametrical axes of the sample up to the cracking point, obtaining the indirect tensile strength.

The conserved resistance value (ITS) is calculated for each sample using the following Expression (1):

$$\mathrm{ITS} = \frac{2\mathrm{P}}{\pi \mathrm{DH}} * 10^3 \quad (1)$$

where ITS corresponds to the indirect tensile strength (kPa), P is the maximum breaking load (N), D is the diameter of the sample (mm), and H is the height of the sample (mm).

This result will be the mean value of the dry and wet samples subset, obtaining the indirect tensile strength ratio (TSR) according to Expression (2):

$$\mathrm{TSR} = 100 * \frac{\mathrm{ITS_w}}{\mathrm{ITS_d}} \quad (2)$$

where TSR corresponds to the ratio of indirect tensile strengths (%), $\mathrm{ITS_w}$ is the average indirect tensile strength of the wet lot (kPa), and $\mathrm{ITS_d}$ is the average indirect tensile strength of the dry lot (kPa).

3. Laboratory Testing Results and Discussion

3.1. Optimal Bitumen Content

This investigation uses the reference mix (REF) without PET as the control mixture. Three samples were made to determine the optimum binder content of the control mixture, with each binder content of 4.5%, 5.0%, and 5.5% by weight of the mixture. The specimens were compacted at 75 blows per side using a Marshall automatic impact compactor. The samples' bulk density and maximum specific gravity were determined according to the

European standard UNE-EN12697-6. The optimum binder content was determined at 5% air voids, obtaining a value corresponding to 5.1% by weight of the mixture.

Table 6 shows the design properties of the control mixture obtained in calculating the optimum binder content.

Table 6. Results of Marshall mixture design of control mixture at the optimal bitumen content.

Parameter	Requirement	Result
Marshall stability at 60 °C, kN	Min. 9	14
Marshall flow, mm	2–4	3.3
Marshall quotient, kN/mm	2–5	3.9
Air voids, %	4–6	5.1
Voids in mineral aggregates, %	Min. 13	16.0
Voids filled with bitumen, %	65–75	74.5
Bulk density, g/cm^3	-	2.390

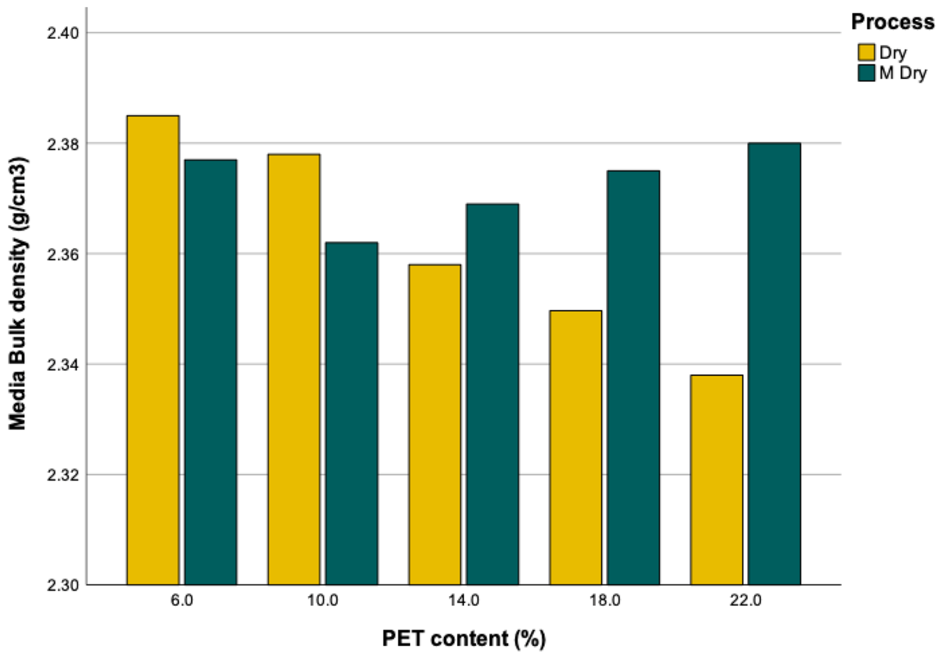
The study used the same binder content (corresponding to the optimum of the control mixture) to manufacture the PET-modified mixtures to facilitate the comparison of the properties between the two.

3.2. Volumetric Parameters of the Mixtures

Figure 4a shows the results of the bulk density of the PET-modified blends as a function of the different contents. A reduction in bulk density is observed for all PET-modified mixtures compared to the control mixture (shown as a solid horizontal line). With the increasing PET content, the bulk density is further reduced. This is because PET has a much lower specific gravity than aggregates, so it will reduce the bulk density when added to the mixture. The (more or less) coarse PET particles result in a relatively high bulk density compared to results published in other investigations using a fine PET. This may be because the bitumen has to coat a more extensive surface when using a finer PET, which will likely result in lower workability during mixing and, therefore, a lower bulk density.

The difference between the bulk density of the blends produced with the two processes is less pronounced with PET contents of 6.0% and 10.0%, while the difference is more significant with higher PET contents (14.0%, 18.0% and 22%). In most cases, it can be observed that the dry process produces a lower bulk density than the modified dry process. Since the same amount of binder (5.1%) is used to manufacture modified mixtures with and without PET, the plastic consumes its share of the binder for coating during mixing. This results in a higher stiffness than the reference mixture, so adding PET will result in a lower bulk density with the same binder content and compaction energy. In the case of the modified dry process, a smaller difference in stiffness can be observed because the coating of the binder on the aggregates has been continued before the addition of PET. This is why the highest bulk density is obtained for the modified dry process if both processes are compared.

The moisture susceptibility of the asphalt mixture can be controlled by a volumetric parameter based on the air voids content. Figure 4b shows the results of air voids in mixtures modified with 6%, 10%, 14%, 18% and 22% PET. It can be observed that there is a higher air voids content when the dry route incorporates PET compared to the modified dry way. This may be because the addition of PET particles before the addition of the binder reduces the workability of the mixture, leading to an increase in the air voids content. However, it can be observed that all PET-modified mixtures comply with the range of 4–6% air voids, except for the mixtures with a percentage higher than 18%, which could be because of the excess PET.



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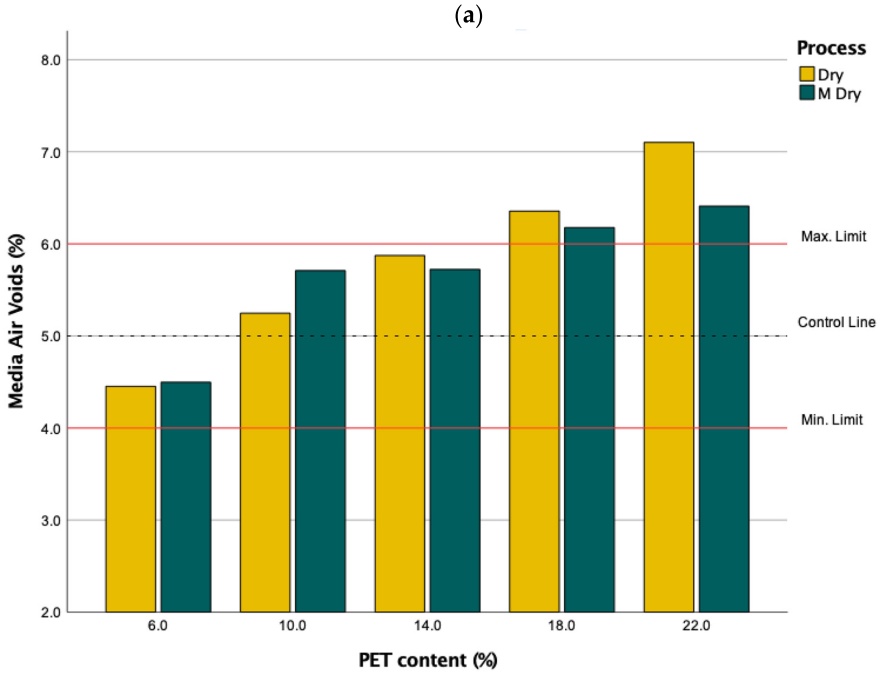

(**b**)

Figure 4. (**a**) Results of bulk density for each % PET sample; (**b**) results of air voids.

3.3. Stability and Flow Marshall

Figure 5 shows the Marshall stability results for the control and PET-modified mixtures. For the mixtures manufactured by the modified dry process, the stability reaches its maximum value at 10% PET, demonstrating that adding PET by the modified dry process increases the stability of the mixture up to a certain plastic content. In the case of the dry process, a constant decrease in stability is observed, being even lower than the stability limited by the regulation. According to the results of other published research, the stability of the asphalt mixture increases with the addition of PET. The stability of the PET-modified asphalt mixture is better compared to conventional asphalt mixture samples due to improved adhesion between aggregate, binder and polymer [30].

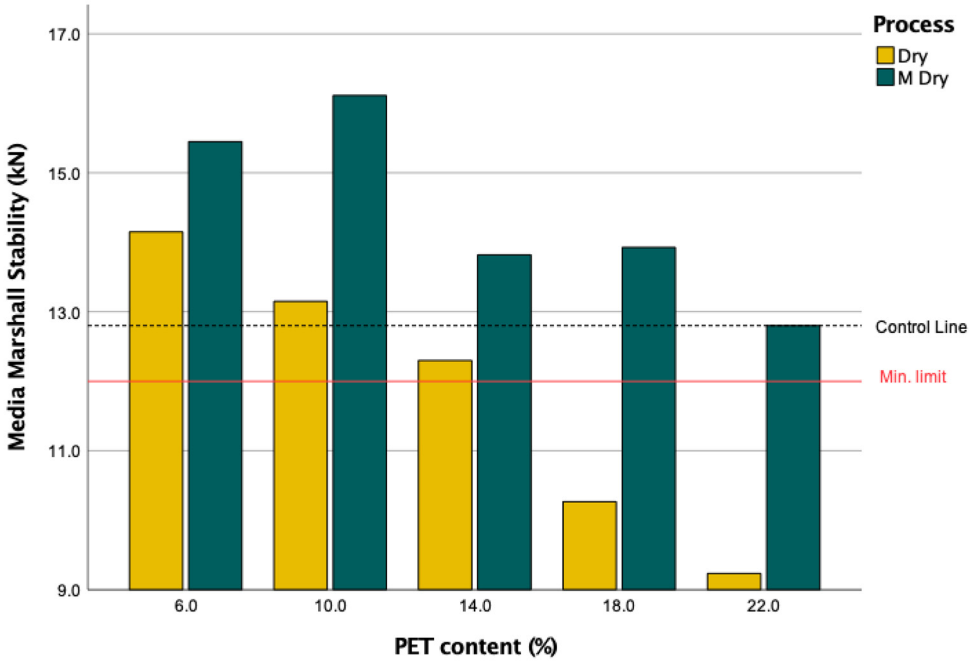

Figure 5. Results of Marshall stability.

Figure 6 shows the Marshall flow results of the PET-modified blends. No clear trend could be discerned concerning the percentage of PET, although all flow values comply with the specified range of 2–4.0 mm. Marginally higher flow values are obtained for the dry modified process than for the dry process. Mixtures with flow values have higher air voids than typical values so the pavement may face premature cracking due to the mixture's brittleness during the pavement's service life. Previous research indicates that the flow increases as the PET content increases [31]; however, it can be observed that in this study, the turning point is at 10% PET and above, as there is an increase in flow for both mixing processes.

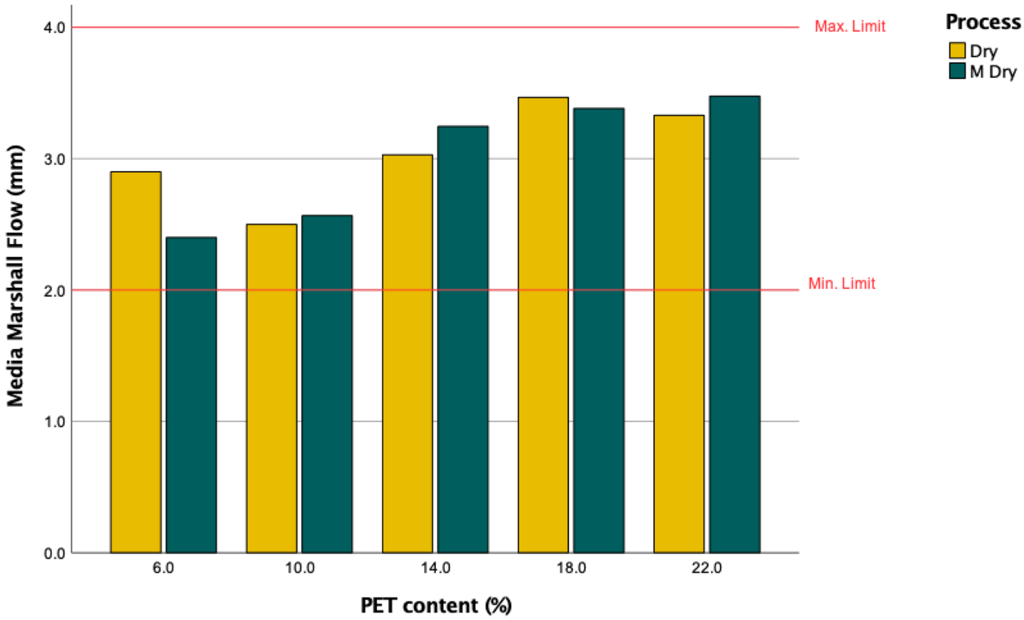

Figure 6. Results of Marshall flow.

The Marshall stability and flow results indicate that the PET-modified mixtures are stiffer than the control mixtures, as they exhibit higher stability and lower flowability. The PET-modified combinations' stiffness is due to PET's semi-crystalline nature, i.e., it presents an amorphous and crystalline structural mixture. This means that above its glass transition temperature (approx. 70 °C), the amorphous part of PET is in liquid form. In contrast, the crystalline part of PET is solid and rigid, as the melting point of PET (approx. 250 °C) is much higher than the mixing temperature (155 °C). The soft proportion improves the bond between the aggregate/binder system, while the rigid crystalline portion adds rigidity to the asphalt mixture.

3.4. Moisture Susceptibility Test

Various tests have assessed the susceptibility of pavements to continuous moisture damage. However, a worldwide standardisation that assesses the degree of water-induced deterioration has yet to be achieved. Because of this, the impact of moisture on asphalt mixtures is assessed based on the ratio of the results obtained between wet and dry samples [32].

This research evaluates moisture susceptibility using the TSR test, defined by the ratio between the indirect tensile strength (ITS) of moisture-conditioned and unconditioned (dry) samples. Figure 7 shows the ITS results conditioned by the moisture content of the PET-modified mixtures.

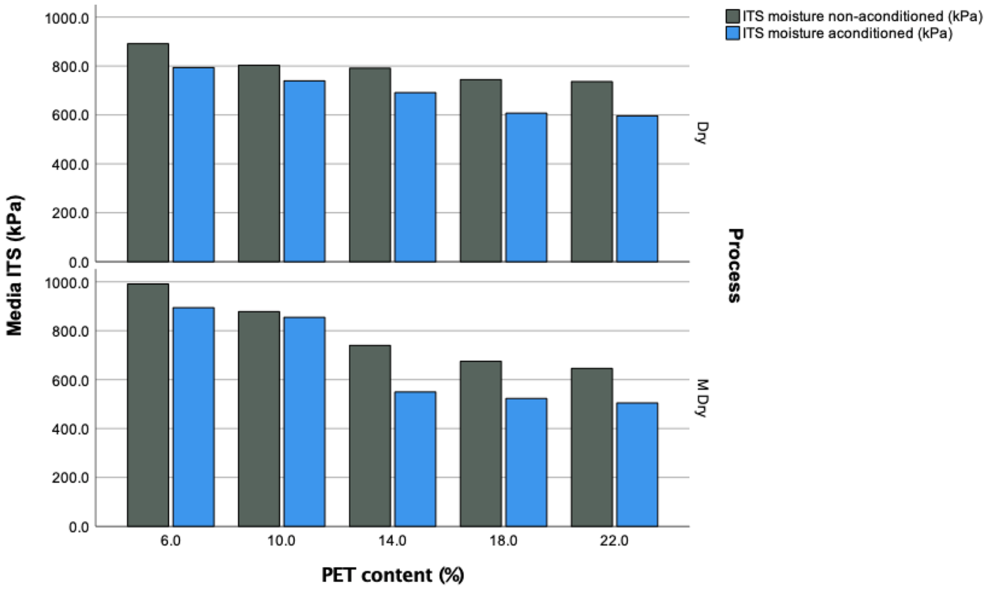

Figure 7. Results of ITS test.

It can be observed that the PET-modified blends show higher ITS values than the control blends up to 10% PET. At a PET content of 10%, the modified dry mixtures show an ITS approximately 20% higher than the dry mixtures. This indicates that the modified dry mixtures resist higher pre-rupture tensile stresses.

Figure 8 shows the TSR values of the PET-modified mixtures with different contents and production processes. It can be observed that for dry samples, the minimum TSR value is higher than 80%, which is adopted by road agency specifications [29]. The mixtures produced by the modified dry process for the percentages of 6 and 10% PET outperform those produced by the dry process regarding resistance to moisture damage. When a higher PET content is used, the opposite effect occurs, i.e., the dry TSR value increases for the mixtures with 14, 18 and 22% PET content, but without reaching the results of the control mixture. According to the results published in other studies, the resistance to moisture damage of PET-modified asphalt mixtures is significantly higher than control mixtures [33,34] which agrees with the results obtained from this test.

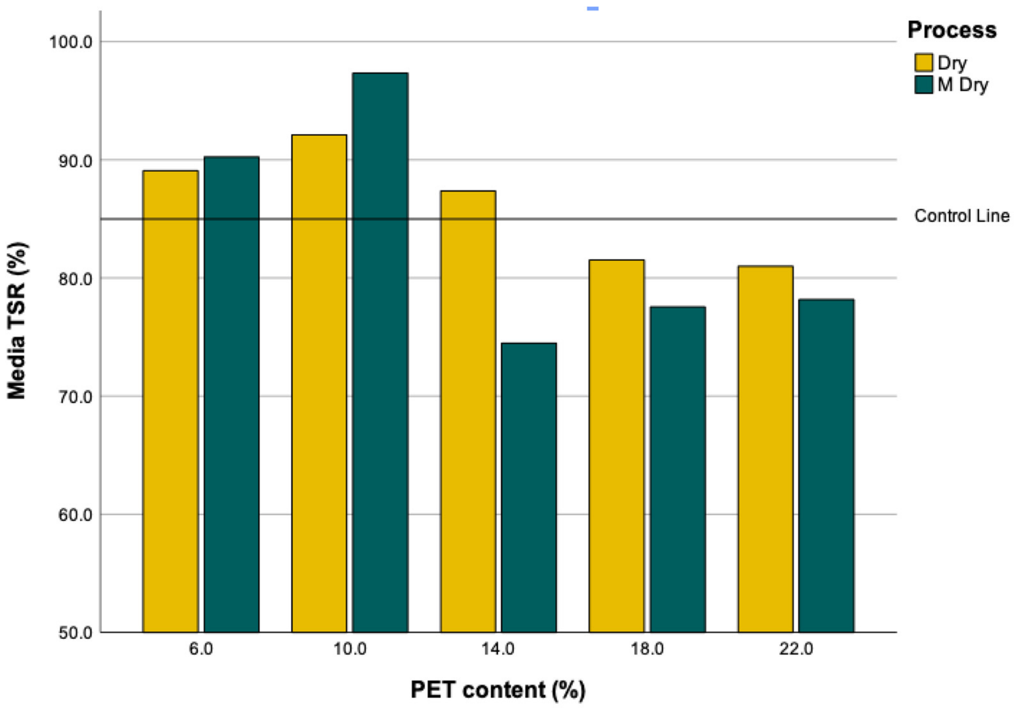

Figure 8. Results of TSR test.

The ANOVA results indicate that the main effects for the PET content are significant, while for the process type there is only significance for Marshall stability (Table 7). The modified dry process produces more moisture-resistant mixtures. The binder coating around the aggregates is likely reduced in the dry process when bitumen and PET are added during the mixture manufacture. This effect is not observed during the modified dry process, as the PET is introduced into the mixture after the aggregates have already been coated with the binder.

Table 7. Results of ANOVA.

Factor	Process Type *p*-Value, S/NS	PET Content *p*-Value, S/NS
Bulk density (g/cm^3)	0.168, NS	<0.001, S
Marshall stability (kN)	<0.001, S	<0.001, S
Marshall flow (mm)	0.840, NS	<0.001, S
TSR (%)	0.325, NS	<0.001, S

3.5. American Standards (ASTM and AASHTO)

A total of 12 test specimens was manufactured in this study, 6 test specimens as reference samples and 6 manufactured with 10% PET (by weight of bitumen). As indicated in Table 5, the American standard classifies the test specimens according to the percentage of air voids present in each sample, with a range between 6–8% for the ASTM standard and 6.5–7.5% for the AASHTO standard.

Figure 9 shows the TSR values of the mixtures manufactured according to the procedures established in the ASTM and AASHTO standards. The results obtained for the ASTM standard indicate that the reference mixtures have a high susceptibility to water, as they do not reach the 75% limit. However, the mixtures with 10% PET substantially improve their resistance, achieving almost 90% TSR, which indicates that the mixture could be applied in wearing courses. According to the AASHTO standard, the results show that the reference mixtures and those modified with 10% PET meet the limit of 80% TSR. However, the reference mixture is the most susceptible to moisture damage.

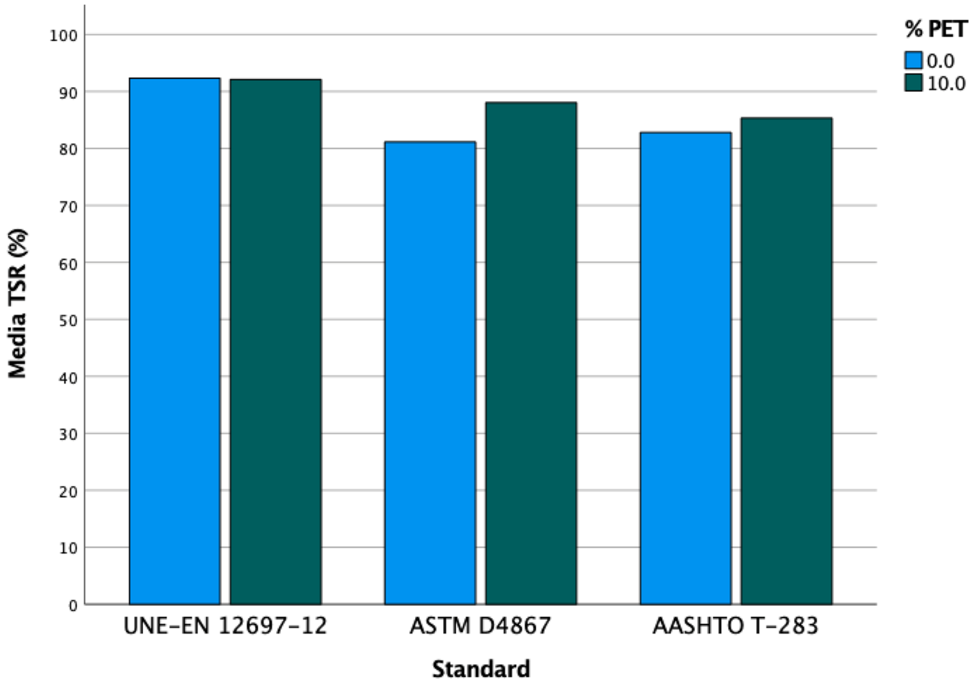

Figure 9. Results of TSR for each standard.

The strength values obtained for the American procedures are below those obtained by the UNE standard. This could be due to differences in test temperature, as there is a difference of 10 °C between the ASTM and UNE standards (from 25 to 15 °C). In addition, the conditioning temperature to which the test specimens are subjected (60 °C) is higher than that of the UNE standard. It is important to note that the lower the temperature is, the higher is the stiffness of the asphalt mixtures.

On the other hand, if the TSR values obtained are related to the PET content by the type of process established in each standard, the results obtained for the European standard are higher than those of the American standards (Figure 10). This indicates that these blends have a higher resistance to the load applied by indirect traction, which is related to the conditioning temperature (the mixtures lose resistance as the temperature increases).

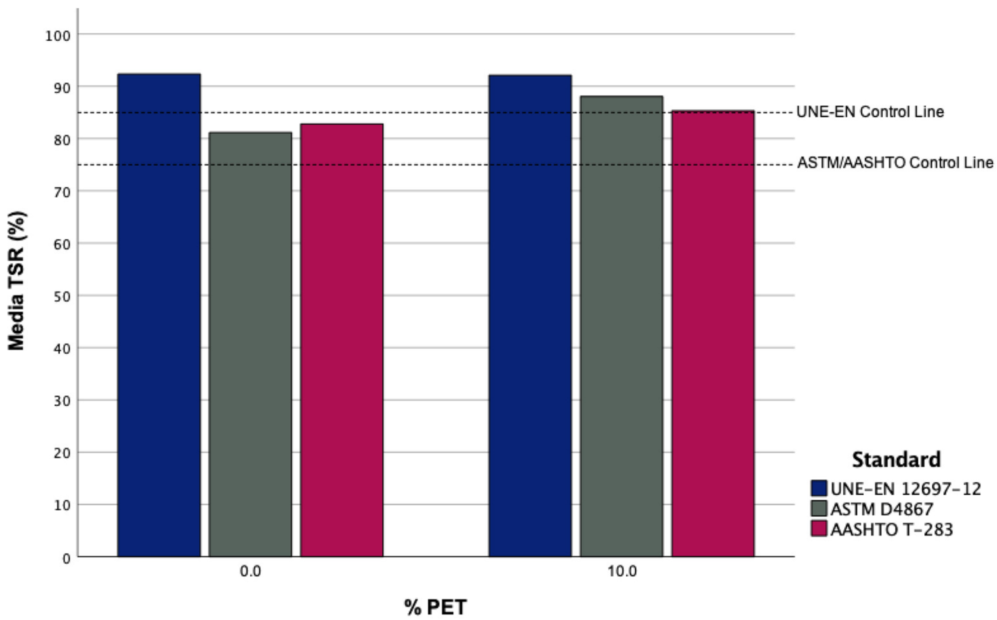

Figure 10. TSR results for each standard.

The results obtained for the reference samples manufactured according to the UNE standard indicate that they are more sensitive to the effect of water than the AASHTO standard. This may seem strange since it would be expected that the samples subjected to freeze–thaw cycles (AASHTO) would present more significant damage due to the effect of water.

For samples made with 10% PET (in general), there are a few differences between dry and wet samples for each standard.

4. Conclusions

Moisture susceptibility is a phenomenon that can cause severe deterioration in asphalt pavements if left unchecked. In this research, the main damage mechanisms associated with the effect of water are analysed based on the tensile moisture susceptibility test (TSR) according to three different standard procedures (UNE, ASTM, and AASHTO) on referential asphalt mixtures modified with a polymeric additive (PET).

1. The use of polyethylene terephthalate (PET) as an additive in asphalt mixtures causes a decrease in the mass-to-volume ratio (density) relative to the reference samples and an increase in the void ratio of the asphalt mixtures.
2. The reference samples (0%) tested according to the UNE standard complied with the limits established for application in surface layers. However, adding 10% PET considerably improved their resistance, reaching 98% TSR. The samples made with 10%, 14%, 18%, and 22% PET did not meet the strength requirements established in the standard and were therefore discarded from further analysis.
3. The samples tested according to ASTM D4867 showed higher moisture susceptibility indices for the reference mixtures (0%), behaving as unstable mixtures under the study conditions. However, with the addition of 10% PET, the mixtures' stability increased, and their susceptibility to water decreased, obtaining results that were within the limits established by the standards.
4. The samples tested according to the AASHTO T-283 standard showed a lower susceptibility to water than those following the previous standard. This is due to the ageing of the samples prior to compaction, where the aggregate achieves a better adhesion with the binder, which in turn improves the indirect tensile strength behaviour of the sample.

This research can be complemented with rolling tests to evaluate the behaviour of PET-modified pavements under the action of vehicle wheels. Using modified dry technology could reduce the additional costs associated with dry mixing of PET in hot mixture asphalt. Because of this, it is important to demonstrate the effectiveness of polyethylene terephthalate and the reactions produced with the aggregate and binder produced by applying the modified dry technology [35]. Finally, this research demonstrates that the effect of water can be a severe problem for pavements and should therefore always be considered at the design stage. The choice of asphalt mixture types can considerably reduce water damage to the pavement structure and increase the pavement's service life.

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