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Modified Asphalt Mixtures Incorporating Pulverized Recycled Rubber and Recycled Asphalt Pavement

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Abstract

In the search to achieve eco-friendly techniques that ensure significant improvements in the properties of hot mix asphalt (HMA), recycled materials are being considered with greater application, coming from the pavement itself and also from artificial elements such as rubber. In this sense, the objective was to study the behavior of the mechanical and microstructural properties of HMA by adding pulverized recycled rubber (PRR) and recycled asphalt pavement (RAP), taking into account a control group without any addition and an experimental group with PRR and RAP. The research involved the production of briquettes with the modification of asphalt cement (AC) using doses of 3%, 5%, and 7% of PRR as a replacement by weight of AC. Then, the optimal percentage of PRR was combined with 10%, 20%, and 30% RAP as a partial substitute for the coarse aggregate. It should be noted that in both aspects, the thermogravimetric and microstructural performance of the asphalt mixture was evaluated. Subsequently, the results obtained indicate that the HMA is MAC-1 type, and it was established that the combinations of PRR and RAP significantly influence the physical-mechanical properties of the HMA with 3%PRR+10%RAP. On the other hand, the findings of the PRR thermogravimetric analysis show that the degradation of HMA occurs at 350°C, causing the loss of both mechanical and microstructural properties. However, infrared spectroscopy and scanning electron microscopy revealed that the PRR adheres correctly with the aggregate, improving the morphology and texture of the HMA.

Keywords: Asphalt; Microstructural Properties; Pavement; Rubber.

1. Introduction

Given the emerging demand, lately the asphalt-producing industry uses high energy consumption to achieve a fluid mixture, in addition to fossil fuels that pollute the atmosphere by the emission of toxic gases [1], hydrocarbons being the most used pollutants that cause respiratory irritation in humans and are used in large quantities for mixtures used in pavements, which implies the indiscriminate depletion of natural resources [2]. The road infrastructure sector in several countries seeks to develop and promote technologies that promote sustainability in pavements through designs oriented to recycling and optimization of resources that achieve an environmentally friendly road development [3]. Countries such as the USA, China, Spain, Indonesia, Japan, and others have chosen to develop alternative studies using reusable waste to improve road infrastructure, carrying out studies of HMA modified with recycled tire rubber, recycled concrete aggregates, coal fly ash, graphene, plastic waste, and others [4, 5]. In addition, countries such as Brazil, Chile, Colombia, and Ecuador replicate the model of these competent and environmentally friendly technologies. Spain, which developed its regulations and manual of Tires out of use (NFU), and Chile, which in 2016 enacted one law about regulation and

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management of NFU, are normative precedents that give relevance to the use of recycled rubber as part of asphalt mixtures applied in new pavements [6, 7].

These alternatives have made it possible to focus on the development of eco-pavements, where the use of pulverized recycled rubber (PRR) and recycled asphalt pavement (RAP) through the production of hot mix asphalt (HMA) stands out. Recycled rubber (RB) is easily obtained in granular and pulverized form, and its temperature, together with HMA, is adequate without losing its properties. In addition, during its production, energy is reduced by 8% to 16%, which means that toxic emissions are reduced by 15% to 57%, and it can be used both wet and dry [8]. RAP is a suitable alternative to replace aggregates in the design of new asphalt mixtures [9], which reduces the consumption of natural resources. A clear example is Europe, which has managed to use 76% of RAP waste in cold and HMA for the resurfacing and construction of their roads [10].

In general, the PRR recycling method is environmentally friendly and is added wet to HMA. Its main physical properties that define its behavior as an asphalt cement (AC) modifier are maximum size, particle size, texture, and temperature. It is obtained through mechanical crushing processes until the desired size is obtained, considering that the finer the particle size, the greater the interaction between AC and rubber [11]. To ensure a higher quality of the PRR particles, it is sieved by passing through the N°20, N°30 or N°40 screen to obtain diameters between 0.25 mm to 1.50 mm, free of textile particles, metallic fibers or other impurities. For its application and heating in the oven, it must present a homogeneous aspect without generating foam, while the approximate temperature to achieve conditions similar to those of an AC is 110°C [12].

The results show that the use of PRR between 0.70 mm and 1.00 mm as AC replacement in an interval of 0.40% to 2.50%, causes a decrease in stability with 0.17%, flow increases 0.19%, the air voids (VTM) in HMA increase in 0.23% and voids in the mineral aggregate (VMA) also increases in 0.16% [13-16]. In addition, with percent between 12% to 25% increased by 0.06%, 0.22% and 0.23%, the stability, flow and VTM, respectively [17, 18]. To achieve an adequate interface, the mixture should not present an excess of rubber-asphalt dosage since it turns it into an insufficient mixture, exposed to plastic deformations in its service period applied in pavements [19]. For the development of new asphalt mix designs with the incorporation of RAP, there are limiting factors for its use that guarantee a quality HMA, such as knowing its type of mix, the graduation of the extracted aggregates, and the determination of its recovered AC [20]. In addition, for further characterization, its properties should be analyzed to know its oxidation and determine the use of rejuvenators [21, 22]. Avoid using RAP with high levels of damage that would affect its composition and an adequate diametral dimension of the particles to avoid oversizing. The idea is to achieve a particle size that is close to the aggregate it replaces and represents a suitable substitute that contributes to the improvement of the pavement where it is applied [23, 24].

European and North American countries use non-renewable waste pavement material as an environmental technique. The United States is one of the countries that, after several investigations, affirms through the National Asphalt Pavement Association (NAPA) that mixtures with high RAP contents between approximately 30% to 40% increase their stiffness and decrease their resistance [25]. Recent research reveals that the substitution of conventional aggregates by recycled aggregates from RAP in the range of 10% to 25% obtains a decrease in flow of 0.02% and 0.03% in VMA but presents increases of 0.02% in flow and 0.07% in VTM. Furthermore, in the 30% to 50% ranges, an increase of 0.07% in stability, 0.34% in VTM, 0.01% in VMA, and 0.03% in flow is obtained [26, 27]. In the case of the combination of asphalt mixtures with the incorporation of PRR and RAP, they developed mixtures with proportions of rubber in relation to the weight of AC in ranges of 12% to 30% and combined recycled aggregates between 30% to 100% and using 12% PRR and 40% RAP, increases in 11.2% by stability and 4.3% by flow were obtained [28]. It was also shown that the inclusion of 100% RAP is not feasible to substitute natural resources, since the ratio of recovered asphalt to conventional aggregates must be restored, and rejuvenators must be used [29].

Most studies focus on the evaluation of the physical and mechanical properties of asphalt mixtures, but few research studies consider studying microstructural characteristics such as homogeneity, morphological changes, and functional groups involved in changes in their macro-structural mechanical behavior, its study being necessary in HMA [19, 30]. The analysis of asphalt mix samples with PRR by scanning electron microscopy (SEM) analysis showed that the rubber particles were dissolved and uniformly adhered to the aggregate particles, which generates changes in shape and roughness [31], whereas Fourier-transform infrared spectroscopy (FTIR) analysis showed that there are more carbon functional groups; this means that the stiffness of the dispersed particles is higher. In addition, due to the increased energy of the asphalt molecules, the rubber absorbs the energy caused by bending vibrations on a flexible pavement application with HMA [32].

2. Material and Methods

This study was based on an experimental investigation of a quasi-experimental type with control sample observations for the application of PRR in order to obtain an HMA with the best dosage and complement it with the incorporation of RAP to optimize its mechanical performance. Figure 1 shows the process flow diagram of the methodology used in this research.

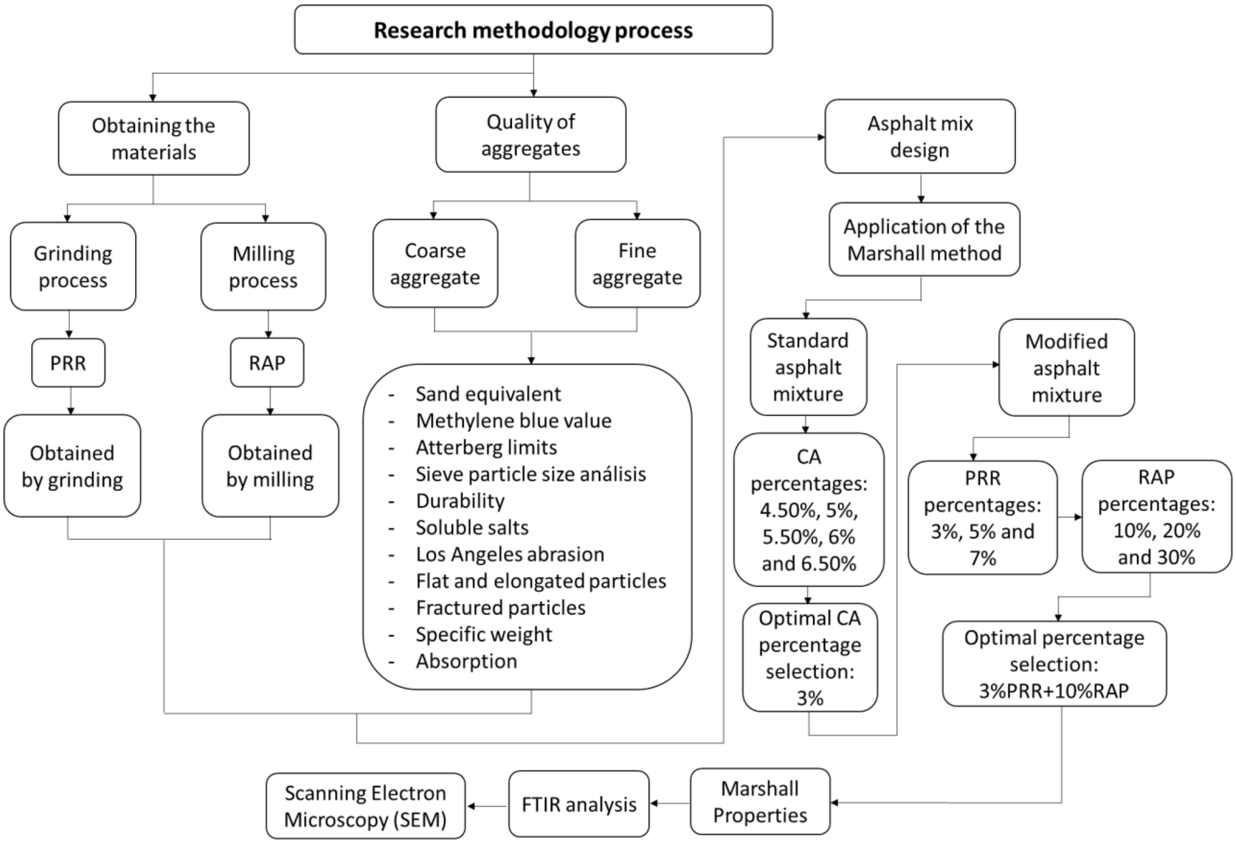

Figure 1. Process flow diagram of the methodology used in this research

2.1. Materials

Natural aggregates from the Tres Tomas quarry, Ferreñafe district, Lambayeque Region, Peru, were used as nominal maximum size (NMS) of ¾" (19 mm). To guarantee the quality of the aggregates, the required specifications and standards were met, whose ranges are shown in Tables 1 and 2. The recycled aggregate used was RAP from the flexible pavement of the airport CAP. FAP. José A. Quiñonez from Chiclayo district, Lambayeque Region, Peru, extracted by the milling technique. The samples acquired had a granulometry control, as shown in Figure 2 and subsequently used in the modified HMA.

Table 1. Physical characterization of natural fine aggregate

Test name	Standard	Specification	Results
Sand equivalent	ASTM D2419-22 [33]	45% Min.	72.00%
Angularity	ASTM C1252-23 [34]	30% Min.	49.90%
Methylene blue value	AASTHO TP 330-22 [35]	8% Max.	6.42%
Plasticity index	ASTM D4318-17e1 [36]	NP	NP
Durability index	ASTM D3744/D3744M-18 [37]	35% Min.	57.10
Adhesiveness (Riedel Weber)	MTC E220 [38]	4% Min.	6.00%
Total soluble salts	ASTM D1888 [39]	0.50 Max.	0.07%
Clay in lumps and crumbly particles	ASTM C142/C142M-17 [40]	1% Max.	0.01%

Table 2. Physical characterization of natural coarse aggregate

Tests name	Standard	Specification	Results
Durability to magnesium sulphate	ASTM C88-99a [41]	18% Max.	7.60%
Abrasion in The Angeles machine	ASTM C131/C131M-20 [42]	35% Max.	17.00%
Adherence	AASTHO T 182-84 [43]	> 95	> 95
Durability index	ASTM D3744/D3744M-18 [37]	35% Min.	53.00%
Flat and elongated particles	ASTM D4791-19 [44]	10% Max.	7.50%
Fractured faces (1 face)	ASTM D 5821-13 [45]	85% Min..	100%
Fractured faces (2 faces)	ASTM D 5821-13 [45]	75% Min.	100%
Total soluble salts	ASTM D1888 [39]	0.50 Max.	0.04%
Absorption	ASTM C127-24 [46]	1% Max.	0.74%

Figure 2. Particle size sample of RAP recycled aggregates

In order to obtain PPR, it was necessary to collect used tires from local workshops that recycle this type of material. These tires were then subjected to a shredding process to separate the other components of the tires, such as steel and fibers. This ensured that the rubber was free of contaminants and external elements. The separated rubber was then subjected to a screening process to obtain the finest particles of the shredded material, which resulted in the production of powdered particles and rubber granules. Finally, PRR previously processed by grinding was used and its granulometry was verified following the recommendations of the Ministerio de Obras Públicas de España [12], whose values are shown in Table 3.

Table 3. Types of PRR according to granulometry

Types of PRR	NMS	Results
P-1	1.50 mm	-
P-2	0.50 mm	0.50 mm
P-3	0.25 mm	-

In addition, its PRR degradation temperature was evaluated by thermogravimetric analysis (TGA) to know the temperature compatible with AC. Type 60/70 AC was used. Its degree of concentration and specifications are detailed in Table 4.

Table 4. Physical characterization of type 60/70 AC

Name test	Specification	Standard	Minimum value	Maximum value	Results
Penetration	To 25°C, 100 gr, 5 seg, 1/100 mm	ASTM D5/D5M-20 [47]	60.00	70.00	67.00
Ductility	To 25°C, 5 cm/min, cm	ASTM D113/D113M-17(2023) [48]	100.00	-	>150
Fluency	Kinematic Viscosity at 135°C, cST	ASTM D2170/D2170M-24 [49]	200.00	-	530.00
Solubility	Solubility in Trichloroethylene, mass %	ASTM D2042-22 [50]	99.00	-	99.80
Volatility	Flash point, C.O.C., °C	ASTM D92-18 [51]	232.00	-	290.00
Density	API Gravity at 60°F, °API	ASTM D70/D70M-21 [52]	-	-	6.80
	Specific Gravity at 60/60 °F				1.02
Thermal susceptibility	Softening Point, °C	ASTM D36/D36M-14(2020) [53]	48.00	54.00	50.00
	Penetration rate	-	-1.00	1.00	0.50
	Mass change, % mass of original	ASTM D2872-22 [54]	-	0.80	0.31
	Retained Penetration, % of original	ASTM D5/D5M-20 [55]	52.00	-	63.00
	Ductility at 25°C, 5 cm/min, cm	ASTM D113-17 [56]	50.00	-	74.00

The process of obtaining the RAP commenced with the identification of areas of worn or damaged asphalt in need of repair or replacement on a local road with medium traffic volume. Subsequently, the deteriorated pavement was subjected to a milling process, whereby the surface layer of asphalt was removed. The material was then transported to a processing plant, where it was temporarily stored prior to undergoing further processing. At the recycling plant, the milled material was crushed in order to recover the aggregates and reach a suitable size. Meanwhile, the recovered aggregates were subjected to particle size treatment in order to separate the particles according to their size, resulting in gravel and sand. Finally, the particles were processed by asphalt washing in order to recover the asphalt binder present in the recovered material.

2.2. Hot Mix Asphalt Preparation

The preparation of cylindrical specimens was developed using the Marshall method to determine their properties. It was carried out in the stages described below. The first was carried out to determine the optimum asphalt cement (OAC) of the HMA mixture using asphalt content at 4.00%, 4.50%, 5.00%, 5.50%, 5.50%, 6.00% and 6.50% as shown in Figure 3. The first was carried out to determine the OAC of the HMA mixture using asphalt content at 4.00%, 4.50%, 5.00%, 5.50%, 5.50%, 6.00% and 6.50%, as shown in Figure 3.

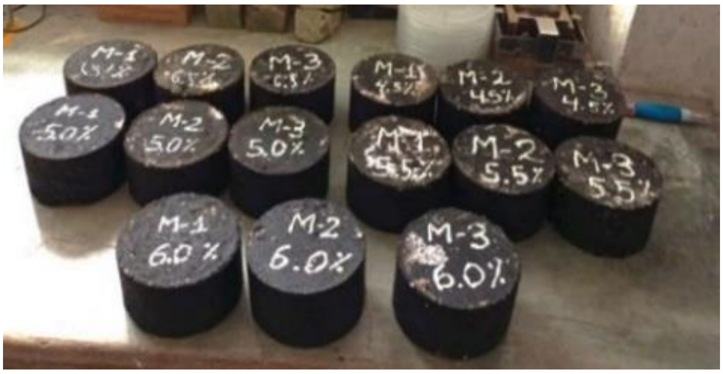

Figure 3. Samples for determining the OAC in HMA

The second was developed from the OAC obtained to determine the Marshall properties of the standard HMA, as shown in Figure 4. The third was based on determining the optimum proportion of PRR in the modified HMA and for its preparation, the OAC obtained was considered and substituted in proportions of 3%, 5% and 7% of PRR in relation to weight, as shown in Figure 5.

Figure 4. Samples of HMA+0%PRR+0%RAP with OAC

Figure 5. Samples of HMA modified with PRR

In the fourth stage, the preparation of the modified HMA specimens with the optimum RAP ratio obtained was carried out, but first the amount of recycled AC was identified by asphalt washing (Figure 6) and then replacing the natural aggregates with recycled RAP aggregates in proportions of 10%, 20% and 30% in relation to weight (Figure 7).

Figure 6. Asphaltic washing of the RAP

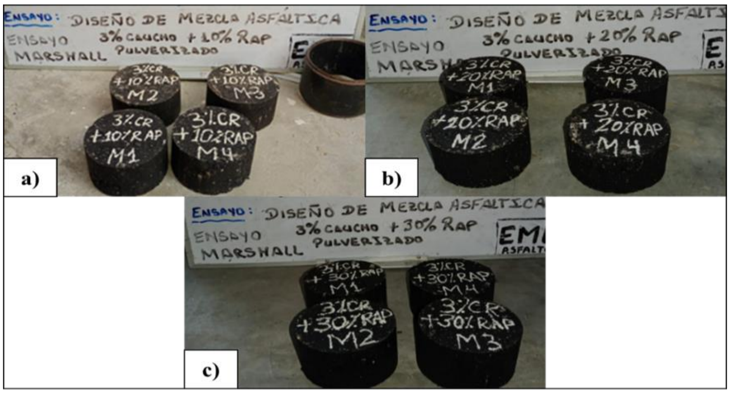

Figure 7. HMA samples with (A) 10%, (B) 20% and (C) 30% RAP

2.3. Microstructural characterization

From SEM with Elemental Analysis (EDS) and FTIR tests, a specimen was generated to extract the core through a 5×5×6 cm cut (Figure 8).

Figure 8. Samples for microstructural studies

3. Results and Discussion

The behavior of HMA with PRR and PRR+RAP was based on analysing the Marshall properties of stability, flow, VTM and VMA.

3.1. Characterization of the PRR

Figure 9 shows that the gradation of the PRR has a linear trend, but complies with the specifications of the Ministerio de Obras Públicas from Spain [12], by developing within the limiting uses, reaching NMS of 0.50 mm, which corresponds to a fine texture, this facilitates greater interaction in the asphalt mix.

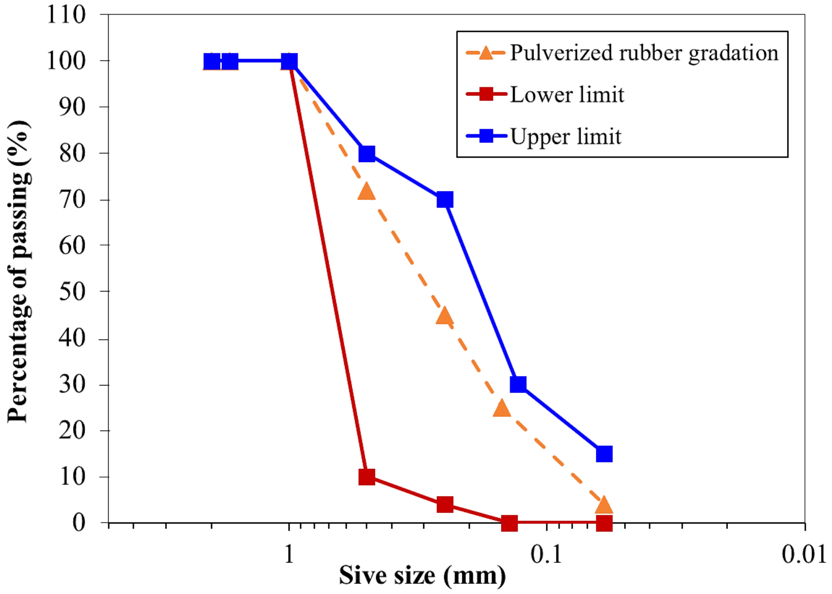

Figure 9. Granulometric curve of the PRR

Figure 9 shows that the PRR gradation has a linear tendency. The particle size curve indicates that in larger regions it approaches the lower limit, indicating an adequate proportion of particles. However, in smaller regions it approaches the lower limit, indicating the existence of a smaller quantity of fine particles. These results comply with the specifications of the Ministerio de Obras Públicas from Spain [12], as they are within the limit values, reaching a nominal maximum size (TMS) of 0.50 mm, which corresponds to a fine texture, which facilitates greater interaction in the asphalt mixture. In addition, they are related to what was specified by Zhuang et al. [19], who indicate that the larger the PRR particle size, the weaker the dispersion effect and the lower the fineness, the interaction of particles, achieving better performance.

Figure 10 shows the thermal degradation of the PRR through TGA, where it loses 3.63% of its initial mass in the range of 50°C to 350°C; in this range, it preserves its properties and is compatible with the temperature of preparation of the AC. Between 400°C to 600°C there is a loss of initial mass of 60%. This does affect performance and practically turns into ashes, which, if combined with the AC, creates a foamy appearance that prevents proper processing interaction in the asphalt mixture that significantly influences its mechanical properties. This result is similar to that obtained by Galeas and Guerrero [57], who state that using PRR with a size of 2.38 mm, its initial mass loss is 0.70% starting at 175°C and indicates that as long as there is no significant thermal degradation, asphalt-rubber interaction will be achieved at a temperature of 110°C to 130°C, which corresponds to the asphalt temperature for an HMA.

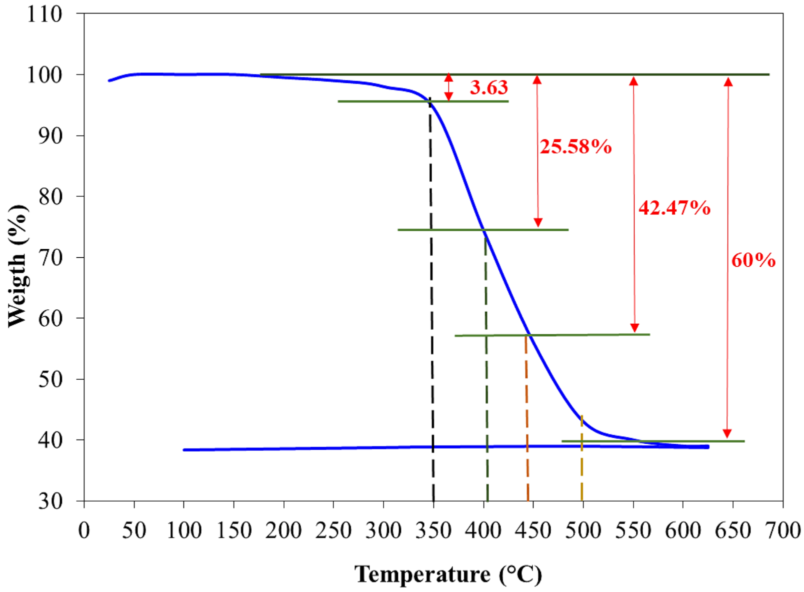

Figure 10. Thermal degradation of the PRR using TGA

3.2. Characterization of Natural and Recycled Aggregates

The results obtained in Figure 11 show that the natural aggregate has an adequate gradation with NMS 3/4", which corresponds to the type of dense mix required for heavy traffic, while the gradation of RAP at the lower limit is slightly different. It indicates that its characteristics are similar to those required, that by combining it with natural aggregates its gradation is adjusted, and by performing the asphalt washing, 4.35 % of recovered asphalt was obtained. The combination of both materials meets the requirements of ASTM C136-06 [58], demonstrating that their application guarantees an HMA mixture of acceptable quality.

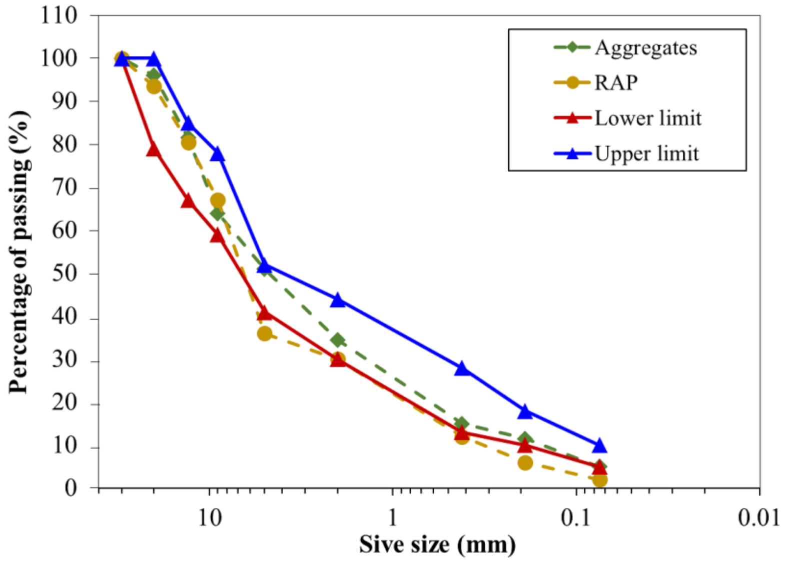

Figure 11. Granulometric curve of natural aggregates and RAP

According to Bocci & Prosperi [28], when the RAP grading is not met, it is due to the low density of its particles, a product of a high content of impregnated asphalt binder or the presence of a high content of fines. For this reason, Fan et al. [29] recommended that the grading should be adjusted with natural aggregates, and asphalt washing should be carried out, where 4.60% of recovered asphalt binder was obtained from a pavement with a useful life of 10 years, while in the present investigation, 4.35% of recovered binder was obtained, corresponding to a pavement with a similar age.

3.3. Marshall Analysis to Determine the OAC

For the determination of the OAC, the parameters were analyzed based on the Marshall method [59], such as: Unit Weight, Stability, Flow, VTM, VMA, VFA and Rigidity, using different concentrations of PEN 60/70 asphalt content, where the results of the HMA indicated that the OAC is 5.76 %, as shown in Figure 12.

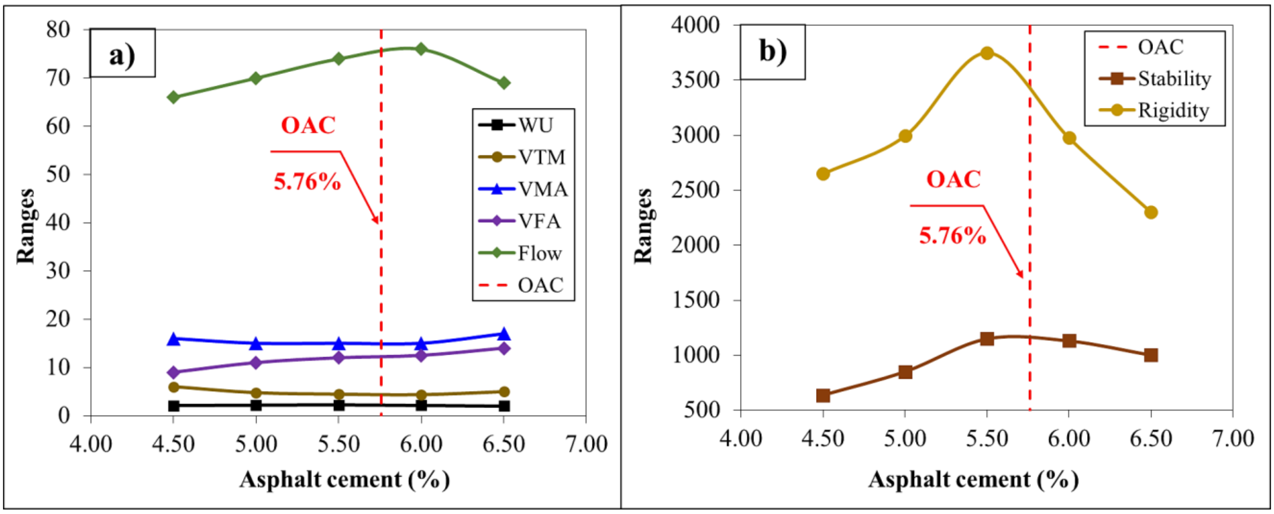

Figure 12. Determination of the optimal asphalt content with PEN 60/70

3.4. Comparison of Standard HMA and HMA with PRR

Percentages were experimented to obtain HMA in order to contrast indicators of stability, flow, VTM, and VMA using the OAC obtained and replacing it according to the 0%, 3%, 5%, and 7% PRR weights, considering the Marshall method, as shown in Figure 13.

Stability refers to the ability of the pavement layer to deform plastically or permanently, and ASTM D1559-89 [59], specifies a minimum stability value of 8.15 kN. Stability values decrease with increasing PRR content but meet Peruvian regulatory specifications for asphalt pavement design. The modified HMA achieves higher stability values with substitution in the range of 0% to 3% PRR, showing an increase of 9.50%, which is shown as an improvement in its strength to heavy traffic loads.

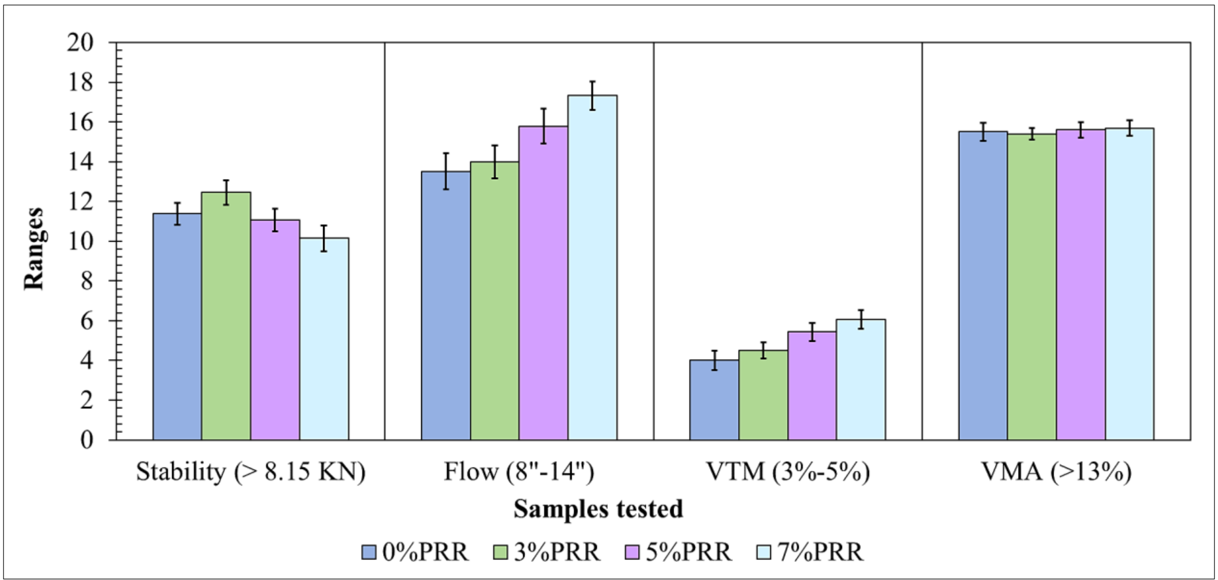

Figure 13. Comparison of variation of the sample standard and sample with PRR

The flow indicates the plasticity of the mixture without cracking, where the ASTM D1559-89 [59] establishes between 8 inches to 14 inches as a limit. It is evidenced that the flow values tend to increase, where high values indicate a mixture with high plastic behavior and low values indicate lower collapse resistance in the mixture. Being that between 0% to 3% HMA with PRR the limits are met and the fatigue resistance capacity of the mixture increases by 3.60%.

The VTM represents the number of voids in HMA, which is limited to 3% to 5% [58]. The VTM value increases with the inclusion of PRR content, reaching a maximum variation of 51.3% with 7% PRR. This effect is related to the reduction of VMA in the material incorporation process. Therefore, when the PRR content is 3%, the VTM value shows an increase of 12.50%, representing a better quality of HMA, as referred to by Al-Azawee et al. [60].

The VMAs are voids resulting from the absence of AC, and the minimum specified limit is 13% according to ASTM D1559-89 [59]. The VMA value increases with PRR proportions of 3% and 7% because the texture contains voids of certain volumes that are not adequately adhered, generating inadequate compaction.

When the PRR ratio is 3%, the VMA value decreases 0.70%, indicating that the compaction quality of the mix is better. Finally, the optimal %PRR that meets the required demands and shows improvements in HMA properties is achieved with 3% PRR.

These results can be compared with the research of Martinez et al. [61] whose results revealed that using 13% recycled rubber grain, using the dry method, generates greater asphalt consumption and reduces stability and flow, while the results of Zhuang et al. [19] indicate that using a range between 5% to 8% of PRR, the properties of the HMA are significantly improved, achieving greater performance, adherence and strength of the pavement against external loads. Likewise, the results obtained by Hittini et al. [16], Olkeba & Potdar [62] and Wang et al. [63], reveal that with doses between 2% to 4% of PRR, good cohesion between the materials is achieved, leading to improved performance of the HMA. However, Zhao et al. [64] indicated that using proportions greater than 5% of PRR, the structure of the asphalt mixture becomes deficient.

3.5. Comparison of Standard HMA and HMA with PRR and RAP

The samples were prepared considering the optimal %PRR obtained and replacing the natural aggregates on a weight basis at 10%RAP, 20%RAP and 30%RAP, using the Marshall method, as shown in Figure 14.

All stability increases achieved by using the experimental %RAP comply with Peruvian regulatory requirements. However, higher stability results were obtained using 3%PRR+20%RAP, with an increase of 23.30%, indicating that the RAP achieves good density levels and is well graded. This allows the PRR to completely cover some voids in the modified asphalt mix. This implies an increase in its mechanical strength, which demonstrates better performance of the HMA, these findings being consistent with the results obtained in the research studies by Tan et al. [13], Zao et al. [14], Chegenizadeh et al. [17], and Sohail Jameel et al. [20].

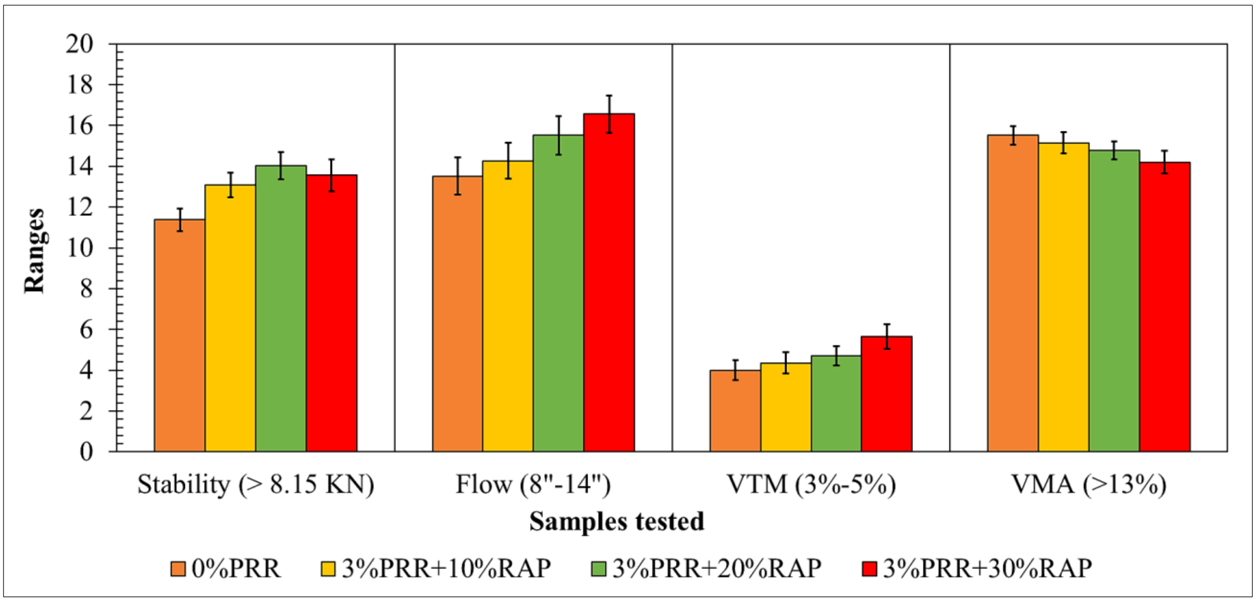

Figure 14. Comparison of variation of the sample standard and sample with PRR+RAP

The results obtained for the flow property increased significantly by 14.80% and 22.60% as RAP incorporation increases, at 3%PRR+20%RAP and 3%PRR+30%RAP, which reached higher values, indicating that they did not meet the regulatory requirements and produced high plasticity mixtures. In contrast, the experimentation of 3%PRR+10%RAP meets the requirement and increases by 5.60%, which shows that the RAP positively influences the density, which, in turn, allows very few voids to exist that are covered with PRR. This generates an effect that contributes to the strength of the asphalt mix exposed to significant deformation from heavy traffic and its durability during its service life.

The VTM value increases with the increase in the proportion of RAP, reaching the requirements with 3%PRR+10%RAP and 3%PRR+20%RAP, indicating that the ideal diametric gradation was present during the RAP incorporation process. This, combined with PRR, fills the void in the mixture, which improves the quality of its application in any flexible pavement. In the combination of 3%PRR+30%RAP, the maximum required limits are exceeded, and significant improvements in the mechanical behavior of the mix are achieved. This is interpreted as the filling of voids significantly influencing the properties of the HMA.

From the above, the VMA results decreased as the RAP ratios increased, meeting the requirements and proving to be less prone to wear because they contain recycled bitumen that seeps between the materials and fills the voids with more bitumen, providing adequate compaction. It can be said that the lower the VMA value, the higher the quality of the HMA.

3.6. Microstructural Analysis

The samples analyzed belong to standard and modified HMA with the optimum percentage of 3%PRR+10%RAP. Using an infrared spectrophotometer, it is possible to identify functional groups and thus confirm the presence of compounds that make up the molecular structure of the bituminous mixture.

3.6.1. Infrared Spectroscopy

The FTIR results show the deformation (strength) behavior of the mixture as the chemical structure changes. Figure 15 shows that both mixtures have the following functional groups: "-C-H", "-C=C-" and "-C-C". The sample HMA with 3%PRR+10%RAP (Figure 15-b) has a higher concentration of carbon molecules, which generates increased vibrational peaks and thus affects the rigidity of the mix asphalt.

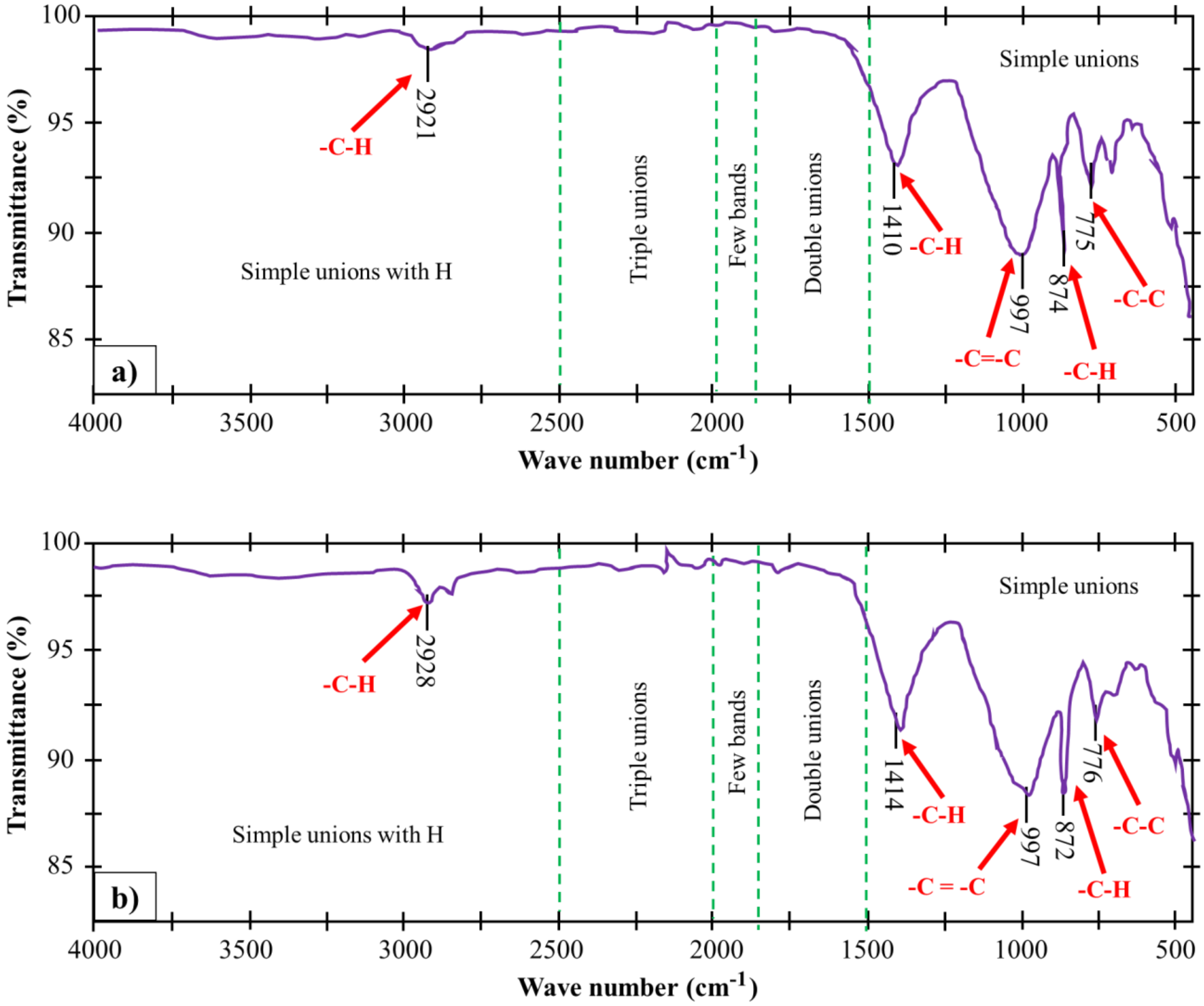

Figure 15. FTIR spectrum: a) sample HMA and b) sample HMA with 3%PRR+10%RAP

Furthermore, it can be observed that the maximum vibrational peak formed by the standard HMA (Figure 15a) is 2921 cm^{-1}, while the modified HMA (Figure 15b) reaches a high peak of 2928 cm^{-1} due to the "-C-H" group, resulting in symmetric stretching and bending. Bending vibration is observed in the peak at 997 cm^{-1} due to the "-C=C-" bonds belonging to alkenes and the commonly formed "-C-C" functional groups.

Likewise, as shown in both cases, the FTIR spectrum according to Figure 15, indicates that the initial region from 500 cm^{-1} to 1500 cm^{-1} is mainly dominated by carbon atoms, dominating each peak, thus forming multiple vibrational modes corresponding to the fingerprint region shown in the assay. These changes are associated with the formation of more aromatic "C=C" rings, indicating the presence of hydrocarbons and asphaltenes that affect the flow of the asphalt mixture, causing a gradual deformation of the asphalt.

These results are related to Meng et al. [65], who infer that these changes are due to the greater presence of the formation of the C=C aromatic ring, which indicates the amount of hydrocarbons and asphaltene, but when using rubber powder, high vibrations are achieved, which provides greater resistance to deformation, while when using rubber grain, various low, dispersed and little visible vibration modes are generated that affect the flow of the asphalt mixtures, leading to the gradual deformation of the asphalt. Similarly, the studies by Nanjegowda & Biligiri [32] and Leiva-Villacorta, & Vargas-Nordcbeck [66] showed that the greater presence of carbon allows higher vibration peaks that generate greater rigidity in the HMA.

3.6.2. Scanning Electron Microscopy and Elemental Analysis

For the characterization of the asphalt mixture, measurements were performed using SEM images at 104x (Figure 16), which allows visualization of the morphology of the area of interest, structure, and presence of craters or pores on the surface of the element. In addition, EDS was performed with an electronic detector, which allows the identification of the chemical substances in the component elements of the sample.

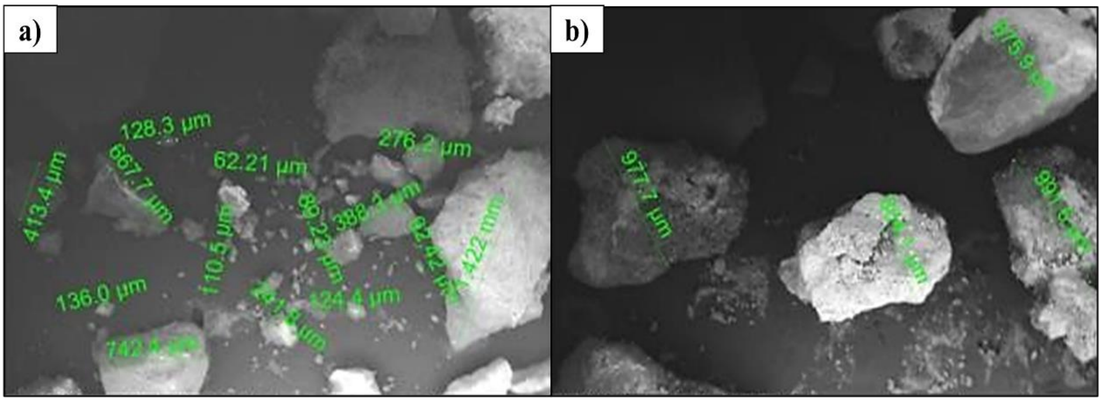

Figure 16. SEM images at 104x: a) sample HMA and b) sample HMA with 3%PRR+10%RAP

Figure 16-a shows the microstructural changes of the pattern HMA, where the aggregate particles present in coarse and fine fragments show that the surface is angular with low sphericity, the appearance is relatively dry, and the particles vary between 99.47µm to 485µm. In addition, it is visualized that the AC surrounds the granular materials, resulting in rugged and crystalline surface textures.

On the other hand, Figure 16b shows the results of HMA modified with 3%PRR+10%RAP. It can be observed that the morphology of the combined aggregates presents a subangular surface with low sphericity, subrounded edges, and a slightly rugged texture, with the presence of wear (cracks) belonging to the RAP aggregates. The aggregate also has a dark impregnation appearance, which corresponds to the combination of AC and RAP, indicating that there is an adequate asphalt-rubber interaction that allows the cracks on the surface of the recycled aggregate to be covered. Finally, a more uniform structure is obtained with a homogeneous appearance and higher capacity for accommodation of materials.

Table 5 shows that HMA has more carbon, which confirms the FTIR results, and also has a measurable oxygen content, but its calcium and silicon components are reduced by 40.24% and 16.20%, respectively. These changes are caused by the presence of hydrocarbons, where the increase in carbon gives the asphalt a higher viscosity, but the oxidation of calcium and silicon occurs, where a higher calcium content provides more resistance to asphalt rutting, improves rutting, and the resistance of the asphalt to low temperatures, reaching a certain degree of homogeneity.

Table 5. Variation of the chemical composition of the samples HMA

Chemical elements	Sample HMA (%)	Sample with HMA with 3%PRR+10%RAP	Variation	Observation
Oxygen (O)	46.27	48.62	5.08%	Increases
Carbon (C)	36.77	39.36	7.04%	Increases
Calcium (Ca)	9.12	5.45	40.24%	Decrease
Silicon (Ci)	7.84	6.57	16.20%	Decrease

These results are similar to those obtained by Zhuang et al. [19] and Vigneswaran et al. [67], since they showed that PRR and asphalt interlock in such a way that their interaction generates an effective dispersion effect, which allows filling voids and non-hydrated areas. In addition, Zhuang et al. [19] highlighted in their results the formation of a complete network in the HMA structure, which allows the mechanical properties to be adequately maintained and the moisture resistance to be significantly increased. On the other hand, the research of Albayati et al. [68], in relation to RAP, revealed that, by SEM analysis, the modified HMA presents a large amount of medium and fine particles, thus generating a rough surface texture, but with a proportional increase in mechanical properties.

3.7. Statistical Analysis of Experimental Samples

The statistical analysis was carried out by applying the complementary randomized design in order to evaluate its level of statistical significance in each of the properties of the asphalt mixtures performed, in accordance with what was pointed out by Bérubé et al. [69] and Gottumukkala et al. [70]. In order to evaluate the significance, assumptions such as: Normality and Homoscedasticity (homogeneity) must be met.

If in the Normality test, the P value is less than 0.05, the null hypothesis is rejected and the significance is evaluated using the Kruskal Wallis Post-Hoc Test; but if the P value is greater than 0.05, the null hypothesis is accepted and the Homoscedasticity Test is performed, where if the P value is less than 0.05, the null hypothesis is rejected and the significance is measured using the Games-Howell Post-Hoc test; but if a P value greater than 0.05 is obtained, the null hypothesis is accepted and the significance is measured using ANOVA analysis.

The significance levels considered are: if $0.01 \leq p < 0.05$, it is considered significant; if $0.001 \leq p < 0.01$, it is considered highly significant; if $p < 0.0001$, it is considered very highly significant; and if $p > 0.05$, it is considered non-significant. The statistical analysis was performed using IBM SPSS Statistics Software and the last combinations of experimental groups were considered, such as: 3%PRR+10%RAP, 3%PRR+20%RAP, 3%PRR+30%RAP compared to the standard sample (0% substitution).

The Stability and VTM properties did not meet the first assumption of normality because the data used are normally distributed, and the Kruskal-Wallis test was applied, which showed that the dosages of 3%PRR+20%RAP, 3%PRR+30%RAP were highly significant, while 3%PRR+10%RAP did not present significance.

The flow property met the first assumption of normality since the data used are not normally distributed. The second assumption of homoscedasticity was met, indicating heterogeneity among the data used. The Games-Howell test was used where the dosage of 3%PRR+20%RAP, 3%PRR+30%RAP is highly significant, while 3%PRR+10%RAP does not show significance.

The VMA property met the first assumption of normality because the data used are not normally distributed. The second assumption of homoscedasticity was met since there is homogeneity among the data used. ANOVA was used; the how was rejected, and by means of Tukey's post hoc test, it was identified that the dosage of 3%PRR+20%RAP, 3% PRR+30%RAP and 3%PRR+10%RAP is highly significant.

4. Conclusions

Based on the results of the investigation of HMA with PRR and RAP influence in comparison with the standard HMA, it is concluded that:

- The use of PRR with a particle size of 0.50 mm and a degradation temperature of 150°C to 160°C, has shown an adequate performance that meets the specifications required to be a partial substitute for AC 60/70; while the coarse and fine aggregates from the RAP have shown a correct homogenization due to the gradation adjustment when combined with the natural aggregates. Both materials are within the granulometric limits, indicating that they are acceptable as partial substitutes for this HMA type.
- To add 3%, 5%, and 7% PRR showed that the ideal substitution ratio in AC is up to 3%PRR because it meets the requirements and Marshall properties performance is improved, where stability, flow, and VTM values increased by 9.50%, 3.60%, and 12.50%, respectively, while VMA decreased by 0.7%, indicating higher resistance of HMA against cracking.
- The results of adding the experimental percentages of RAP to the optimum mixture of PRR-modified HMA indicate that the best effect occurs with 3%PRR+10%RAP, where stability, flow, and VTM are improved by 15%, 5.60%, and 8.70%, respectively, while VMA decreases by 2.40%, which indicates a greater effect on the performance becoming a mixture with higher rutting resistance and high dispersion capacity under loads applied in intense vehicular traffic.
- The microstructural results show that between the standard HMA and that modified with 3%PRR+10%RAP, the PRR is able to adhere adequately to the AC, within the temperature range applicable for the preparation of HMA, covering the natural aggregate and cracks with RAP; in addition, it is evident that its morphology and texture are improved with the AC percent optimum, which provide higher viscosity to the asphalt.
- Finally, the incorporation of PRR for HMA with RAP has a positive and significant effect on the performance of the mechanical properties of HMA, reaching with 3%PCR+10%RAP a very significant influence; therefore, according to the findings of our research, the application of this type of HMA would be viable and sustainable for flexible pavements.

5. Declarations

5.1. Author Contributions

Conceptualization, M.C. and E.C.; methodology, W.B.; software, R.S.; validation, M.C., E.C., and G.A.; formal analysis M.C.; investigation, W.B.; resources, E.C.; data curation, G.A.; writing—original draft preparation, M.C.; writing—review and editing, G.A.; visualization, R.S.; supervision, M.C. and G.A.; project administration, R.S.; funding acquisition, R.S. All authors have read and agreed to the published version of the manuscript.

5.2. Data Availability Statement

The data presented in this study are available on request from the corresponding author.

5.3. Funding

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5.5. Conflicts of Interest

The authors declare no conflict of interest.

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