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Influence of the content and particle size of rice husk ash on the water susceptibility of asphalt mixtures

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ABSTRACT

This study investigates how rice husk ash (RHA) content and particle size influence the water susceptibility of asphalt mixes. A two-factor experiment was conducted, varying RHA proportions (2.5%, 5.0%, and 7.5%) and particle sizes (149, 74, and 53 µm). The RHA, sourced from agro-industrial wastes in Trujillo, Peru, was previously analyzed by, scanning electron microscopy (SEM) showed irregular, porous, and angular particles, typical of ashes produced at moderate temperatures, which enhance binder interaction and internal friction. The ash analysis also identified crystalline phases like cristobalite and quartz, suggesting chemical reactivity. Coarse and fine aggregates met Peruvian standards for durability and wear resistance. The optimal asphalt cement content was 4.39%, balancing cohesion, stability, and air voids. The experimental results indicate that adding 5.0% RHA, especially with particle sizes smaller than 53 µm, significantly improves the mixture's resistance to moisture damage. This not only enhances the longevity of pavements but also promotes sustainability by incorporating waste materials into road construction. The incorporation of RHA not only improves the technical performance of asphalt mixtures, but also represents an environmentally beneficial strategy by reusing an abundant agricultural waste, reducing the environmental impact and promoting a circular economy.

Keywords: asphalt mixtures; water susceptibility; rice husk ash; stability; TSR Test.

1. INTRODUCTION

The incorporation of rice husk ash (RHA) into asphalt mixes has received much attention as a sustainable and environmentally friendly alternative to traditional fillers. RHA, a by-product of rice husk combustion, offers a promising solution to reduce the environmental impact and carbon footprint of asphalt production while improving the mechanical properties of mixes.

Currently, climate change and global warming are a global concern, generating debates in both the public and scientific spheres. In this context, the road pavement industry recognizes the importance of adopting more sustainable alternatives in the design and production of asphalt mixtures (Valdés-Vidal et al., 2020).

The use of RHA in asphalt mixtures contributes to resource conservation by reducing the demand for natural aggregates and mineral filler materials. This procedure helps preserve natural resources for future generations and reduces the environmental impact associated with mining and quarrying activities (Jwaida et al., 2024) (Mistry et al., 2023). Furthermore, RHA is a cost-effective alternative to traditional fillers, as it is obtained from waste materials that are readily available and economical. The use of RHA in asphalt mixtures can generate significant cost savings in pavement construction, making it an attractive option for sustainable infrastructure development (Mistry et al., 2023; Maha et al., 2022).

Asphalt pavement derived from nonrenewable crude oil (Lim et al., 2024) has been widely used in road engineering because of its favorable properties, such as flexibility, skid resistance, stress dissipation capacity, and noise and dust reduction (Guo et al., 2020; Hussein et al., 2023). However, over time, exposure to vehicular traffic and weather conditions, such as frequent rains, causes pavement wear, generating unevenness, potholes, and material detachment. In addition, poor drainage prevents the evacuation of moisture, which contributes to localized saturation of the pavement and favors the formation of potholes and the loosening of asphalt (Kim & Le, 2023; Sarkar & Elseifi, 2023).

Traditional pothole repair techniques, such interim fixes and cold patching, are frequently ephemeral because they break down in bad weather and with repeated traffic loads. Therefore, in order to guarantee more sus-

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tainable road maintenance, it is imperative to create materials with enhanced moisture resistance and durability (Lee & Le, 2024).

A key element in this context is the asphalt mix, a material composed of asphalt as a binder, aggregates, and voids (Hayder et al., 2018). Asphalt, obtained by distilling petroleum, is an essential component in the construction of roads, highways, and airports (Bastidas-Martínez et al., 2024), as the performance of asphalt mixtures depends to a large extent on the bitumen used (Sajadi et al., 2025). However, asphalt is a material that is sensitive to temperature, and variations in temperature can have a significant impact on its viscoelastic characteristics (Liu et al., 2023).

Recent advances in asphalt pavements include the incorporation of polymers to improve performance, reduce rutting, reduce susceptibility to water, and mitigate cracking (Kosma et al., 2017; Zhu et al., 2023; Vargas & Hanandeh, 2022). These polymers are thicker than regular asphalt binders and stick better to the materials used in the pavement, leading to thicker layers that resist damage from air and help the asphalt last longer (Bassheet & Latief, 2025; Ye & Zhao, 2023).

The susceptibility of asphalt pavements to moisture degradation is being investigated by researchers worldwide (Valentin et al., 2021). This degradation increases the development of ruts, fractures, and potholes by decreasing the pavement's stiffness and load-bearing capacity (Jweihan et al., 2023; Zarroodi et al., 2023). Water leaking into the pavement surface affects the adhesion between the aggregates and the binder, which eventually leads to asphalt loosening.

The cohesiveness and adherence of the asphalt mixture's particles must be sufficiently enough to keep them from separating when water is present in order for it to be moisture-resistant (Peyman, 2016). According to Cao et al. (2023), the asphalt binder's acid number has a significant role in the mixture's cohesiveness, and moisture damage often happens more around bigger aggregate particles than around the filler (Antunes et al., 2015). Beginning with a negative pressure brought on by vehicle loads, this phenomenon causes pumping, which gradually erodes the asphalt-aggregate interface and speeds up their dissociation. Asphalt mixes' mechanical and physical qualities deteriorate due to the spread of microcracks and weakness at contact areas, which can result in water damage and other issues on the pavement surface. Failures in asphalt cohesiveness and adherence to aggregates may result from such deterioration (Adwar & Albayati, 2024).

Because acidity is frequently associated with SiO_2 content, using mineral fillers might reduce the sensitivity of asphalt mixtures to temperature and moisture fluctuations, particularly if the fillers aren't very acidic (Valentin et al., 2021). The performance of the pavement is also influenced by the particular gravity, size, porosity, texture, shape, and particle size distribution of the filler. Performance of pavement is also influenced by the size, texture, shape, porosity, specific gravity, and particle size distribution of the filler (Zangooeinia et al., 2023).

To guarantee the mixture's impermeability, a sufficient quantity of compacted bitumen must be present (Alireza & Rezvan, 2021).

Li et al. (2020) found that biomaterials such as rice husk ash had both hydrophobic and hydrophilic components that alter the chemical characteristics of asphalt binder. Moreover, these biomaterials enhance the rigidity of the binder and augment adhesion with aggregates. Shenyang et al. (2021) discovered that the quantity of SiO_2 in aggregates and their roughness significantly influence the water retention capacity of the asphalt-aggregate system.

Ash from rice husk combustion is a residue generated as a result of the incineration of this agricultural by-product (Kumar et al., 2022). Husks, which constitute the external coating of the rice grain and are obtained during the milling process, represent the total weight of the grain (Hidayat et al., 2023). Globally, its generation is considerable, given that about 20% of world rice production—which exceeds 545 million metric tons per year—corresponds to this residue (Chilaka et al., 2022). Due to its substantial caloric value of around 3281.6 kcal per kilogram, rice husk has emerged as an alternative energy source, particularly in thermal applications such as brick production (Liou et al., 2023). The material is processed at controlled temperatures between 700 and 900 °C to produce RHA (Zangooeinia et al., 2023). At these temperatures, organic components like cellulose, hemicellulose, and lignin are nearly entirely decomposed, resulting in a silica-rich ash with characteristics applicable in several industrial sectors.

In the specific case of Peru, the Ministry of Agrarian Development and Irrigation of Peru (2024) has identified the region of La Libertad as one of the most important in terms of agroindustrial production. There, rice is positioned as the second most important crop in terms of harvested area, reaching 30 thousand hectares and generating close to 300 thousand tons of product annually. However, despite this high production volume, the management of the resulting husk remains inefficient. A minority of it is used as fuel in industries such as the brick industry, while most of it is burned near the milling centers without taking advantage of its energy value or its potential as a reusable input.

There are several advantages of using agro-industrial and industrial waste, including rice husk ash, to make asphalt mixtures (Trevizan et al., 2023). These include preserving natural resources, lessening the influence on the environment (Camargo-Perez et al., 2024; Raj et al., 2022), and cutting down on building expenses and the carbon footprint (Ram & Ramakrishna, 2022) Accordingly, it was shown that while adding rice husk ash reduces indirect tensile strength, it increases Marshall stiffness. Additionally, higher dynamic modulus and creep values were noted. However, the mechanism of action of these additions remains unclear or insufficiently studied, which limits their application. Therefore, we need to further analyze their microstructure and performance characteristics (Zhu et al., 2024).

By lowering expenses and encouraging a more environmentally friendly approach, the utilization of industrial waste materials, such as rice husk ash, not only improves the qualities of bitumen and asphalt but also produces financial gains (Zangooeinia et al., 2023).

The indirect tensile strength ratio (TSR), which gauges the mixture's resistance to moisture damage, was one of the most pertinent tests used in this investigation to assess the moisture resistance of asphalt mixes. An increase in TSR values indicates lower sensitivity to moisture and higher stability of the asphalt to water damage. The likelihood that the asphalt binder will disintegrate in the presence of moisture was also assessed using other useful tests, such as water immersion tests and Marshall stability (retained stability index). These tests make it possible to evaluate the quality of the adhesion from the beginning of the process (Al-Saffar, 2024).

In order to enhance pavement design for better performance in inclement weather, the primary objective of this study is to determine how the quantity and size of rice husk ash impact the amount of water that asphalt mixes can absorb. Along with determining the ideal proportion of asphalt cement in the mixture, the study aims to describe the physical and chemical characteristics of the ash employed. As a consequence, the outcomes will not only further our understanding of civil engineering but also offer useful recommendations for the use of sustainable and alternative materials in the building of road infrastructure.

2. METHODOLOGY

2.1 Object of study

The target universe were asphalt mixes, while the sample universe corresponds to asphalt mixes with rice husk ash as a filler. The sample consists of an asphalt mix destined for roads in the province of Trujillo, in the Department of La Libertad, Peru, for which briquettes with a diameter of 101.6 mm and a height of 63.5 mm were prepared, adjusted to the optimum percentage of asphalt cement.

2.2 Research design

were prepared, ensuring the repeatability and reliability of the results in the water susceptibility tests. This study used an experimental methodology with a two-factor factorial design. The amount of rice husk ash (RHA) and its particle size were modified to examine their impact on water susceptibility. The independent variables were evaluated at three levels (2.5%, 5.0%, and 7.5% RHA; 149, 74, and 53 µm particle size), resulting in a total of nine experimental combinations. For each combination, three replicates plus three patterns were produced, giving a total of 30 briquettes compacted using the Marshall method. This replication ensured the reliability of the results and allowed statistical analysis to be applied to validate the differences observed.

2.3 Study variables

The present research had 3 variables (two independent variables and 1 dependent variable). The independent variables were the amount of RHA, with three levels (2.5%, 5.0%, and 7.5%), and the particle size, with values of 149, 74, and 53 µm. We evaluated the susceptibility to water, the dependent variable, based on the different levels of the independent variables.

2.4 Experimental procedure

The experimental procedure began with the acquisition and preparation of the materials. Coarse and fine aggregates were obtained from the Bauner S.A. quarry in Trujillo, Peru; rice husk ash from the Ceramirex brick production plant in Trujillo, Peru; and asphalt cement from Chemimax S.A. in Lima, Peru.
RHA was analyzed by X-ray diffraction (XRD) to identify its crystalline structure. The aggregates were examined using tests for particle size, wear resistance, weight and water absorption, plasticity limits, sand quality, resistance to sulfates, soluble salts, broken surfaces, flat and long particles, and the methylene blue test. After that, the mix design was improved using a formula from the Asphalt Institute to find the best amount of asphalt cement, which was changed based on briquette tests to get the right void ratio. The asphalt mix was made by drying and sorting the aggregates, heating them with the asphalt to certain temperatures for mixing and compacting, and then shaping the compacted samples using the Marshall hammer.
In the stage for testing water susceptibility, the specimens were divided into dry and conditioned groups; the conditioned group went through a process of vacuum saturation, freezing, thawing, and heating to simulate moisture damage. Finally, the indirect tension test was done to check how strong the specimens were and to find out the stress-to-retained-stress ratio (TSR), which helped assess how well the asphalt mixture handles water. For a better understanding of the procedure, see Figure 1, which presents a summary of the experimental procedure.

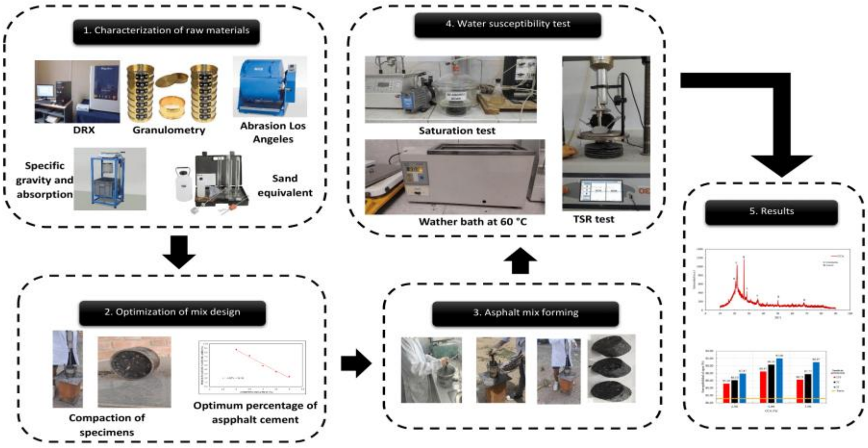

Figure 1. Graphical summary of the experimental procedure

3. RESULTS AND DISCUSSIONS

3.1 Research design

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Table 1. Semiquantitative chemical analysis

Chemical Compound	SiO_2	Al_2O_3	CaO	SO_3	Fe_2O_3	MgO	Na_2O	K2O	P_2O_5	Cl	TiO_2	SrO	MnO	LOI
%	61.13	6.89	0.62	1.19	1.44	0.53	0.37	1.72	0.37	0.15	0.28	0.01	0.08	25.2

The X-ray fluorescence (XRF) characterization used in this work is based on the data reported by Alvarado et al. (2023).

The morphology of rice husk ash (RHA) was analyzed by scanning electron microscopy (SEM), observed at a magnification of 2500×, with an accelerating voltage of 20 kV and a field of view of 83.0 μm (Figure 2). The image shows particles with irregular shapes, angular edges and rough surfaces, with a heterogeneous size distribution. This morphology is typical of partially amorphous ashes obtained by combustion at moderate temperatures without atmospheric control, as in brick kilns. The porous and angular texture of the particles favors interaction with the asphalt binder, which can improve adhesion, increase internal friction and, consequently, contribute to greater structural stability of the mixture, in addition to reducing susceptibility to moisture damage.

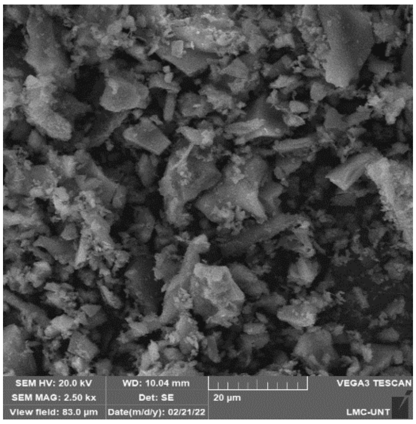

Figure 2. Microstructure of RHA studied at 2500×

Figure 3 shows the results of the X-ray diffraction (XRD) analysis of the RHA, which identified cristobalite (C) and quartz (Q) as the main crystal types, indicated by strong peaks at certain angles, suggesting that part of the material was exposed to high temperatures during combustion. However, this finding seems contradictory in light of the high loss on ignition value (25.2%), which indicates the presence of organic material and suggests that not all of the mass reached sufficiently high temperatures uniformly. This duality is common in combustion processes with poor thermal control, such as brick kilns, where both crystalline phases and a significant amorphous fraction with high pozzolanic potential are formed. The X-ray diffractogram of RHA indicates the presence of dominant crystalline phases, specifically cristobalite and quartz, identified by diffraction peaks at $2\theta \approx 21.9°$ and $2\theta \approx 26.6°$, respectively. The formation of cristobalite confirms that the sample underwent high temperatures during calcination, aligning with the transformation of silica to this phase at elevated temperatures. Conversely, quartz, indicated by the peak at $2\theta \approx 26.6°$, is characteristic of the silica phase typically found in calcined plant-derived materials (Muhammad et al., 2019). The low-angle region confirms the presence of an amorphous fraction in the sample, which is common in ashes subjected to controlled combustion without achieving full crystallinity. This amorphous fraction exhibits high pozzolanic reactivity, enhancing the potential of RHA as a cementitious material.

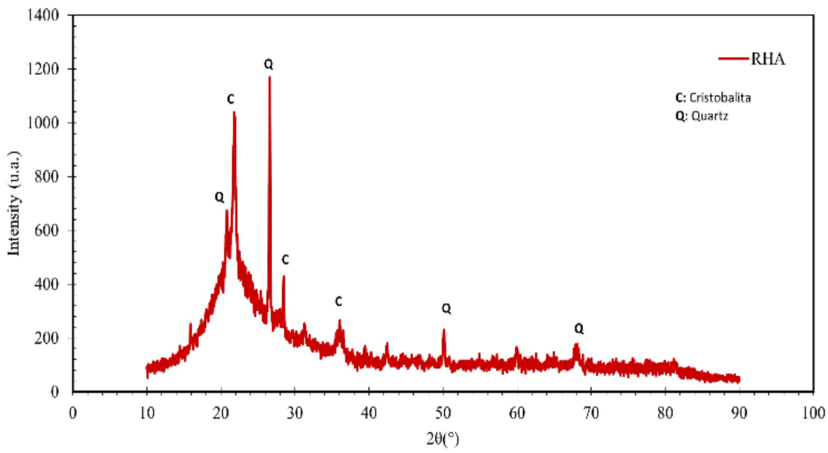

Figure 3. XRD diffractogram of RHA

The particle sizes of the RHA were obtained by dry mechanical sieving, using laboratory sieves with standard ASTM mesh openings (No. 100, 200, and 270), corresponding to 149, 74, and 53 µm, respectively. This particle size classification allowed the specific effect of each fraction on the water susceptibility of the asphalt mixtures to be evaluated.

3.2 Aggregate characterization

The results of the tests conducted on coarse and fine aggregates to assess their characteristics in accordance with defined criteria are presented in Table 2. All tests meet the established criteria, thereby underscoring the material's appropriateness for incorporation into asphalt mixtures. The strength and durability of the finished mix are influenced by the quality of the coarse and fine aggregates, thus the results of assessing their mechanical and physical characteristics in accordance with Ministry of Transport and Communications of Peru (2013) regulations are crucial.

For coarse and fine aggregates, the durability test using magnesium sulfate yielded results of 6.47 percent and 11.62 percent, respectively, both falling within the 18 percent limit, indicating a good resistance of the aggregates against disintegration by the action of aggressive agents. In pavements exposed to unfavorable weather conditions, this outcome is crucial for preventing premature failures. The coarse aggregate's Los Angeles abrasion value of 20% is less than the permitted limit of 40%, indicating its high wear resistance, which keeps the mix stable and lessens surface damage from heavy traffic.

The content of flat and elongated particles was 6.5%, complying with the maximum 10% requirement. This measurement is relevant since particles of unfavorable geometry can negatively affect the workability and compaction of the mix, while a good distribution and shape of the particles improve the density, which contributes to greater strength and durability of the pavement.

The percentage of fractured faces, 0.23%, is much lower than the limit of 1.7%, showing that the aggregate has a shape that helps it stick to the asphalt binder; this feature enhances the mix's internal strength and its ability to withstand pressure and sliding from vehicles.

The total soluble salts in the coarse aggregate were 0.35% and in the fine aggregate were 0.23%, both below the 0.5% limit, which is important to stop internal reactions that could weaken the pavement; having low soluble salts helps prevent issues like efflorescence or damage from harmful chemical reactions.

For water absorption, the coarse and fine aggregates had values of 0.44% and 0.46%, respectively, both under the 0.5% limit, indicating that the aggregates can absorb water in a controlled way, which helps avoid moisture problems in the mix that could weaken it when exposed to water for a long time.

Conversely, the sand equivalent, which is 88.10%, exceeds the minimum requirement of 60%. This indicates that the mix is lacking in detrimental fine material, such as clays, which could potentially compromise its cohesion and adhesion.

Furthermore, the methylene blue test result of 3.7 % is below the 8% limit, indicating that the asphalt mix is both durable and strong due to the low quantity of harmful clay and the high level of cleanliness. Ultimately, the plasticity index was determined to be "NP" (non-plastic), which is advantageous due to the potential for excessive plasticity to compromise the mix's capacity to withstand repeated pressure.

Table 2. Aggregate characteristics

TEST	COARSE AGGREGATE	FINE AGGREGATE	REQUIREMENT	OBSERVATION
Magnesium sulfate durability	6.47	11.62	18 % máx.	Complies
Abrasion angels	20	-	40 % máx.	Complies
Flat and elongated particles	6.5	-	10 % máx.	Complies
Fractured faces	0.23	-	1,7 %	Complies
Total soluble salts	0.35	0.23	0.5 % máx.	Complies
Absorption	0.44	0.46	0.5 máx.	Complies
Sand equivalent	-	88.10	60	Complies
Methylene blue	-	3.7	8 % máx.	Complies
Plasticity index	-	NP	4 % máx.	Complies

3.3 Determination of the optimum asphalt cement percentage

The inverse relationship between the percentage of asphalt cement and the percentage of air cavities in the asphalt mix is illustrated in Figure 4.

As the asphalt cement content increases, the percentage of air voids decreases, which is essential to ensure a compact, well-cohesive mix. This adequate void level helps reduce permeability and minimizes exudation problems, improving the pavement's fatigue resistance, stability, and durability, which is key to prolonging the service life of the pavement in service conditions.

Determining the optimal amount of asphalt cement is crucial to ensure the right performance of the asphalt mix, as it affects its mechanical qualities and durability.

This study employed the trend equation derived from the relationship between asphalt cement content and air voids: y = -3.2857x + 18.438, as illustrated in Figure 3. By substituting y = 4%, we derived an optimal value of 4.39% asphalt cement. This number generates a composition with an optimal distribution of voids, mitigating problems such as water infiltration and oil seepage, while ensuring effective adhesion of components, hence enhancing the pavement's durability, stability, and longevity.

An appropriate asphalt cement percentage facilitates adequate compaction, diminishing the likelihood of ruts and water damage; however, an excessive or insufficient percentage may compromise the pavement's lifespan by permitting moisture or air ingress, accelerating its deterioration.

The standard mix consisted of 44.02% coarse aggregate (gravel), 55.98% fine aggregate (sand), and 4.39% asphalt cement, corresponding to the previously determined optimum percentage. No additional mineral filler was incorporated into this base mix. In the modified mixtures, rice husk ash (RHA) was added as a supplementary material in proportions of 2.5%, 5.0%, and 7.5%, based on the total weight of the mixture. This addition did not replace any component but was considered an additional percentage of the standard mixture. The void volume in the mineral aggregate (VMA) of the standard mixture was 13.35%, with a void volume filled with asphalt (VFA) of 72.33%. In the mixtures with RHA added, the VMA varied between 12% and 17%, while the VFA ranged between 72% and 80%, which shows that the incorporation of ash altered the internal structure of the mixture, affecting asphalt retention and compaction density in a manner dependent on the content and particle size used.

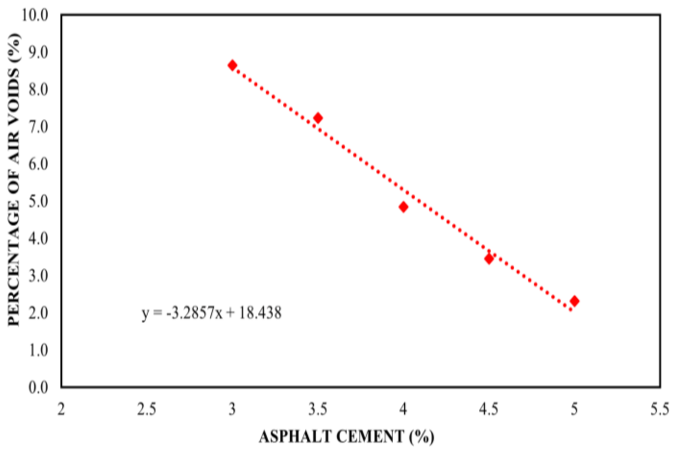

Figure 4. Percentage of air voids as a function of asphalt cement content.

3.4 Susceptibility to water

Figure 5 illustrates the variation in water sensitivity of asphalt mixtures with varying proportions of RHA (2.5%, 5.0%, and 7.5%) and particle sizes (150, 75, and 53 µm), in relation to a standard reference value. The horizontal axis denotes the quantity of RHA added, whilst the vertical axis signifies the degree of water susceptibility, expressed as a percentage. As particle size diminishes, water susceptibility escalates across all studied quantities of RHA, reaching a maximum at a particle size of 53 µm. In contrast, as the quantity of RHA increases, water susceptibility escalates, reaching a maximum at 5% RHA across all studied particle sizes. This evidence indicates that the size of the particles and the amount of RHA added affect how asphalt mixtures react to water, which is important for making them last longer in wet conditions.

This data indicates that increasing water susceptibility is advantageous until it attains an optimal level (5.0%

RHA), beyond which additional modifications may cease to benefit the mixture. Moreover, it is noted that smaller particle sizes, such as 53 µm, exhibit greater susceptibility to water than bigger sizes, indicating that finer particles enhance the mixture's responsiveness to water.

The pattern illustrated in the figure can be comprehended by examining the impact of RHA particle size on the asphalt mixture; smaller particles, such as those measuring 53 µm, possess a larger surface area. The increased surface area facilitates superior integration of RHA particles with asphalt cement and promotes uniform interaction with aggregates, resulting in a more robust bond that reduces water entry. RHA enhances this susceptibility owing to its distinctive physical and chemical characteristics. When amalgamated with asphalt cement, the ash can create a denser and less permeable structure, so enhancing the cohesion of the asphalt mixture and augmenting its water resistance. The dimensions of the ash particles are critical; diminutive RHA particles integrate more efficiently into the interstices between the aggregates. The filling effect, along with the development of supplementary bonds between the cement and ash, improves material adhesion and decreases water absorption, thus rendering the mixture more robust and less susceptible to moisture damage.

However, when the RHA content goes above 5.0%, the mixture becomes less dense and compact because too many particles create more gaps (segregations), allowing more water to be absorbed, which makes it more vulnerable. This phenomenon happens because having too much RHA creates more spaces for water to get in or makes the mixture less firm, reducing its ability to resist moisture.

When comparing our results with those of Mahto et al. (2024), which similarly utilized rice husk ash, it is evident that in both investigations, the RHA improves the properties of the mixture, notably its cohesion and moisture resistance. In this work, we see that the particle size of RHA significantly influences its behavior, with smaller particles, such as those measuring 53 µm, being more susceptible to water due to their larger surface area, which enhances their integration with asphalt cement and adhesion. This study demonstrates a more pronounced effect of RHA particle size, indicating that smaller particles, such as those measuring 53 µm, are more influenced by water due to their increased surface area, which facilitates superior integration with asphalt cement and enhances adhesion. Conversely, the research conducted by Deb et al. (2023) demonstrates that the use of rice husk ash markedly enhances the characteristics of asphalt mixtures, resulting in improved mechanical properties.

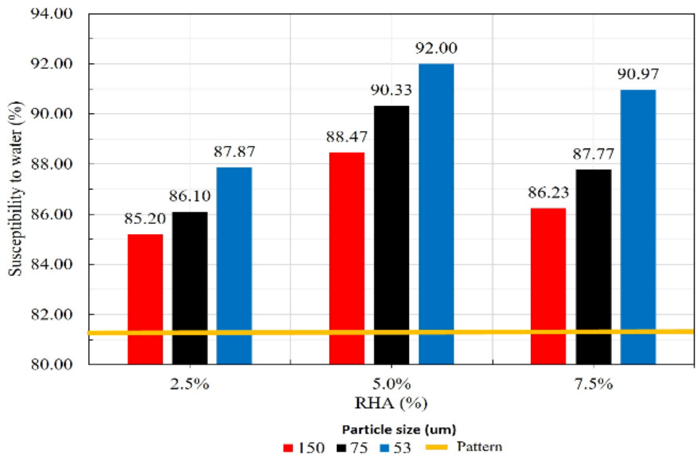

Figure 5. Water susceptibility of asphalt mixtures as a function of RHA content and particle size.

4. CONCLUSIONS

This study confirmed that rice husk ash (RHA), derived from agro-industrial waste, can be effectively used as an additive in asphalt mixtures. It was observed that both the quantity and size of RHA particles directly influence moisture resistance. In particular, the addition of 5% ash with a size of 53 µm offered the best results, improving the internal cohesion of the mixture and increasing its resistance to water damage. The mixtures modified with RHA showed variations in their volumetric properties: the VMA ranged between 12% and 17%, and the VFA between 72% and 80%, compared to the values of the standard mixture (VMA of 13.35% and VFA of 72.33%). These changes reflect how ash interacts with asphalt cement and aggregates, promoting better binder distribution and greater compaction when fine particles are used. In addition to the technical contribution, the use of RHA also represents an environmentally responsible option. Taking advantage of this agricultural waste helps reduce the use of traditional materials and supports more sustainable practices in road construction. In contexts where the aim is to improve pavement durability and reduce environmental impact, RHA appears as a practical, economical alternative that is aligned with the principles of the circular economy.

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