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Experimental evaluation of high RAP bituminous mixtures modified with recycled waste plastics

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ABSTRACT

The increasing demand for sustainable road construction materials, driven by environmental concerns and resource scarcity, has accelerated efforts to incorporate recycled components into bituminous mixtures. In this context, reclaimed asphalt pavement (RAP) and recycled plastics have emerged as promising materials, which may be able to enhance both the environmental and mechanical performance of road pavements. While the individual effects of RAP and recycled plastics have been extensively studied, their combined influence still needs to be further explored. The present study investigated bituminous mixtures containing a recycled plastic compound (RPC) derived from waste plastics and 50 % RAP through a comprehensive laboratory testing program, which included determination of their stiffness properties, cracking resistance, and anti-rutting potential. The role of binder type was also examined by combining RPC with either a softer binder or a harder binder with a bio-based rejuvenator. For comparison, two additional mixtures were produced and tested: a control mixture produced with a neat bitumen without any modification and a mixture containing a SBS highly-modified binder. An extensive statistical analysis was performed on test data to evaluate the actual differences in mechanical characteristics among the mixtures. Overall, the experimental findings demonstrated that the combined use of RPC and high content of RAP represents a viable and promising solution for the production of sustainable asphalt mixtures. When employed together with a suitably selected neat binder and a rejuvenating agent, the modification with RPC may deliver performance levels comparable or even exceeding those of mixtures incorporating a SBS highly-modified binder.

1. Introduction

The growing global emphasis on environmental stewardship, resource efficiency, and infrastructure resilience, is driving the demand for more sustainable road construction materials. As infrastructure development accelerates worldwide, natural resources are increasingly strained. As a result, the road construction sector faces the dual challenge of minimizing environmental impacts while ensuring cost-effectiveness and long-term durability.

Traditional road pavement construction practices rely heavily on virgin materials, such as aggregates and bituminous binders, which contribute significantly to ecological degradation and carbon emissions [1]. To address these issues, the asphalt industry is progressively adopting circular economy principles, with a particular focus on reducing the use of virgin materials in bituminous mixtures and promoting the integration of recycled alternatives [2].

Among recycled materials, reclaimed asphalt pavement (RAP) is the most widely utilized, offering both economic and environmental benefits [3]. RAP is primarily obtained by milling aged pavement layers, with smaller quantities sourced from wasted asphalt plant mix [4]. It can replace more than 50 % of virgin aggregate in new hot-mix asphalt (HMA). Although full recycling (100 % RAP) is technically achievable, most national standards and production practices limit RAP content to approximately 60 % or lower due to concerns related to binder aging, workability, and mixture homogeneity. Its incorporation in road paving mixtures has been shown to reduce greenhouse gas emissions by approximately 17 % and lower the overall environmental footprint of road construction projects by 15–30 % [5–9]. Additionally, incorporating RAP into both bound and unbound layers contributes to reductions in energy and water consumption, while also mitigating

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environmental impacts such as acidification, eutrophication, and human health risks by up to 20 % [10].

Despite the advantages highlighted above, the use of high RAP contents in HMA remains limited, primarily due to concerns about the durability and cracking performance of aged binders [11,12]. Different countermeasures have been adopted to address these issues, including the use of softer binders and the addition of rejuvenating agents [13]. Historically, the use of softer binder grades has been the most widely adopted strategy. However, this approach presents notable limitations at higher RAP contents, such as a diminished impact on the final binder properties [14]. Additional challenges include logistic constraints at the asphalt plant, which may require an impractical number of bitumen storage tanks when managing multiple RAP sources and varying RAP percentages [15]. The use of rejuvenating agents represents an effective alternative in high-RAP mixtures; several studies have indicated their ability to restore the properties of aged binders, enabling bituminous mixtures with high RAP content to match or even exceed the performance of conventional virgin mixtures [16–21]. In addition to the effects on physical properties of aged binders, rejuvenators can also partially restore the chemical functionalities lost during oxidative aging, improve blending and aged binder reactivation, and thereby enhance the long-term durability of high-RAP mixtures [22–24]. The use of recycled plastics in bituminous mixtures has attracted growing attention, driven by the dual objectives of improving pavement performance and addressing the escalating global plastic waste crisis [25]. Research has demonstrated that recycled plastics can serve as effective modifiers for mixtures, with performance largely influenced by the chemical composition of the plastic and its dosage [26,27]. In general, plastic-modified bituminous mixtures exhibit increased stiffness and improved resistance to permanent deformation, which is particularly beneficial in high-temperature regions and under heavy traffic loads [28–30]. However, higher dosages may lead to excessive stiffness and potential cracking issues [31].

Plastics are commonly incorporated into bituminous mixtures through two different processes, referred to as dry and wet processes. The dry process entails adding plastics with high melting point directly into the mixing batch, in substitution of one or more aggregate fractions. Several types of plastics have been investigated by researchers, including polyethylene (PE), polypropylene (PP), polystyrene (PS), and polyethylene terephthalate (PET) [32,33]. These studies reported that mixtures containing plastics added via dry process exhibited improved mechanical properties, with material cost savings up to 10 %; however, increasing plastic content above 5 % generally led to reductions in stability and density of the mixtures, partly jeopardizing the environmental benefits associated with recycling. The wet process involves blending plastics with a low melting point into the bituminous binder to produce polymer-modified bitumen (PMB). The effects of several types of plastics on the mechanical performance of mixtures, in terms of stiffness, resistance to permanent deformation, and fatigue life, have been investigated [34–38]. These studies collectively highlight the effectiveness of the wet process on laboratory mixture performance using various recycled products, although issues related to potential phase separation between plastics and binder may arise. Moreover, the wet process requires more machinery and equipment to shred the plastics into powders and mix them with hot bituminous binder, thus resulting in a more energy- and time-consuming process [39–44]. However, it should be noted that, to date, the incorporation of recycled plastics via wet methods has mainly been demonstrated at laboratory or pilot scale, with industrial-scale applications still limited.

Recognizing the strengths and limitations of both dry and wet methods, researchers have proposed a hybrid (or mixed) process that combines elements of both approaches. This strategy aims to optimize the interaction between plastic waste, aggregates, and binder to enhance both mechanical performance and environmental benefits. Specifically, the hybrid method involves introducing low melting point plastics (typically used in the wet process) directly into the mixing batch during asphalt production, following a procedure similar to that of the dry process [45]. Some authors [30] investigated the addition of high-density and low-density polyethylene (HDPE and LDPE, respectively) up to 10 % by weight of the binder, reporting contrasting effects on fatigue and rutting resistance depending on the plastic type and binder characteristics. Lastra-González et al. [46] demonstrated that plastics recovered from copper cables and flexible packaging improved stiffness and rutting resistance. Similar performance trends were also reported by Eskandarsefat et al. [47], who evaluated a graphene-enhanced plastomeric bitumen modifier. Conversely, Abdalfattah et al. [48] reported improved anti-rutting performance but noted a reduction in cracking resistance at intermediate temperatures. Angelone et al. [49] incorporated recycled silo bags containing LDPE, HDPE, and PP at very high dosages of 2 %, 4 %, and 6 % by weight of the total mixture, concluding that the 2 % content offered optimal improvements in stiffness and thermal susceptibility without adversely affecting flow, tensile strength, or moisture resistance. Poulikakos et al. [31] confirmed that the inclusion of PE waste enhanced mixture stiffness, cohesion, and resistance to permanent deformation, identifying an optimal content of 1.5 % by weight of the total mixture. However, higher PE dosages were associated with increased rutting susceptibility under wet conditions.

These findings underscore the viability of using plastic waste as a sustainable and effective modifier, provided that material type and dosage are carefully optimized. Overall, research demonstrates that incorporating recycled plastics via dry or mixed methods can achieve performance comparable to or superior to conventional bituminous mixtures, without introducing additional environmental burdens [39]. Moreover, the hybrid method offers operational advantages over the wet process, particularly in addressing storage and stability challenges. Unlike the wet process - which typically requires specialized agitation systems and stabilizing agents to maintain binder homogeneity - the hybrid approach incorporates plastics during the aggregate mixing phase, eliminating the need for additional equipment or additives. This simplification reduces manufacturing costs and improves production efficiency [32,40].

While the individual effects of RAP and recycled plastics have been widely investigated, a relatively limited number of research studies have examined their combined influence, especially in those mixtures with high content of RAP.

Some investigations have examined mixtures containing 30 % RAP and recycled plastics used as aggregate substitutes via the dry process. These investigations reported improvements in moisture resistance, indirect tensile strength, fatigue life, and rutting resistance compared to control mixtures without plastics [50–52]. Leng et al. [53] introduced PET-derived additives via ammonolysis into high-content RAP mixtures using the wet process, resulting in enhanced softening point, rutting resistance, and fatigue cracking resistance, while enabling the co-recycling of RAP and plastic waste. Riccardi et al. [54] explored the combination of polyacrylonitrile fibres with RAP contents up to 50 %. They found that the addition of polyacrylonitrile fibres worsened the moisture susceptibility resistance, but improved the rutting, stiffness properties, fatigue performances, and cumulative fatigue damage of the mixtures with and without RAP. La Macchia et al. [55] investigated the performance of mixtures incorporating 50 % RAP and a polymeric compound derived from waste hard plastics added via the hybrid method. Based on the experimental results obtained from testing, they emphasized the importance of optimizing plastic content to ensure acceptable materials performance.

Further research is clearly needed to complement previous findings and to support eventual large-scale applications. In this context, the experimental work presented in this paper aimed to provide a contribution to research by evaluating the performance of bituminous mixtures produced with 50 % RAP by total aggregate mass and a commercial recycled plastics compound (RPC) derived from waste plastics. The novelty of the study lies in combining high RAP content with RCP using the hybrid method, across various RPC dosages and different types of virgin binders added to the mixture.

Moving beyond the work of La Macchia et al. [55], which focused primarily on base properties of materials for mix design purposes, the present research was performance-oriented. To this regard, a comprehensive set of laboratory tests was conducted on the considered mixtures to provide a broader understanding of their mechanical response and to assess their suitability for use in road pavements. For comparison purposes, two additional mixtures were produced and tested: a control mixture without any modification containing only paving grade bitumen to assess the effect of different RPC contents, and a PMB reference mixture to compare the performance of the wet and hybrid modification methods. In this second reference mixture a polymer modified bitumen designed and produced for asphalt mixtures with elevated RAP content was used. This binder fulfils requirements set in national standard ČSN 65 7222–3 [56] and is referred to as PMB RC.

The laboratory tests were selected to determine relevant performance-based parameters commonly used for the characterization of mixtures. They included stiffness properties through four-point bending (4PB) tests, rutting resistance through wheel tracking (WT) tests, crack propagation resistance and fracture toughness through semi-circular bending (SCB) tests, and low-temperature flexural strength through three-point bending (3PB) beam tests. Fatigue analysis was not included in this study; however, relevant findings on fatigue properties of mixtures containing 50 % RAP and recycled plastics have been reported in another paper by La Macchia et al. [57].

The analysis of the results was conducted with the specific goal of highlighting the combined effects of recycled plastics and RAP on mixtures' performance as a function of the variables under consideration.

2. Materials

The bituminous mixtures investigated in this study were produced by combining various base materials, including five fractions of virgin aggregate, a filler, a single-size RAP, three types of bituminous binders, an RPC, and a rejuvenating agent.

Virgin aggregates and filler were sourced from different quarries located in Czech Republic: they included spilite (Zbraslav quarry), chert and gneiss (Lašovice quarry), and limestone filler (Velké Hydčice quarry). The RAP material, collected from a local supplier, was of 11RA 0/8 type, with a nominal content of aged binder equal to 5.1 % by total mass.

The particle size distribution curves of virgin aggregate fractions and filler are shown in the diagram of Fig. 1. The diagram also shows the particle size distribution of the RAP material after being subjected to binder extraction using the rotary evaporator method according to EN 12697–3 [58].

The bituminous binders included two neat bitumen and one highly SBS PMB. The neat binders (Binder A and Binder B) were characterized by different penetration grades and were used to produce bituminous mixtures containing RPC. Following the recommendations of Czech technical standards [59], the softer Binder A was utilized to produce mixtures without the addition of a rejuvenating agent, due to its ability to soften the aged binder of RAP; conversely, the harder Binder B was utilized in combination with a rejuvenating agent, to mitigate stiffening effects due to RAP. Binder A was also utilized to produce a control mixture without any modification. The SBS PMB (Binder C) was used to prepare an additional reference wet-modified mixture; it contained approximately 6 % virgin SBS polymer with a specific formulation tailored for high-RAP contents. The description and characteristics of all considered binders are reported in Table 1.

The RPC consisted of selected recycled plastics deriving from post-consumer and post-industrial waste. According to the processing scheme adopted by the manufacturer, plastic waste underwent washing, separation, grinding, and pelletizing operations to obtain polymer granules with a diameter of 4–6 mm. An industrial patent protected the final product; it mainly consists of PP and PE, even though the exact formulation remained proprietary and confidential. The main characteristics of the RPC are summarized in Table 2.

The granules of the RPC were added to other mix components directly into the mixing batch. According to the definition provided by Giustozzi et al. [63], the dry modification of asphalt mixtures requires the use of polymers with a softening temperature at least 30 °C higher than the mixing temperature, which is typically set at 170 °C. Since the

Table 1
Description and characteristics of bituminous binders according to [60,61] and [62] for neat and SBS highly-modified binders, respectively.

Binder code	Binder type	Penetration at 25 °C (dmm)	Softening point (°C)
Binder A	Neat bitumen 70/100	81	46.2
Binder B	Neat bitumen 50/70	56	53.4
Binder C	SBS highly-modified bitumen (PMB 25/55–65 RC)	46	78.6

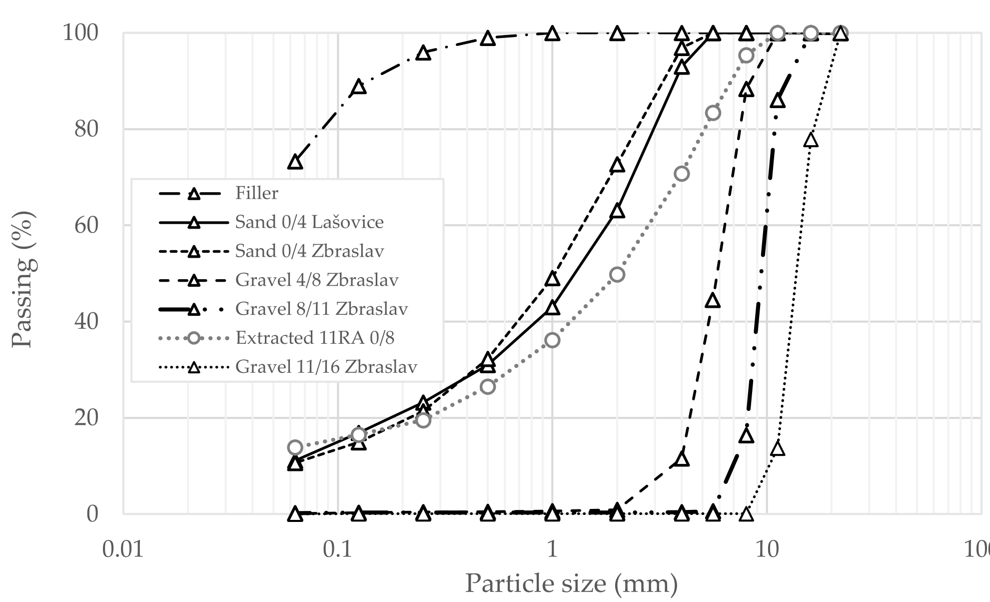


Fig. 1. Gradation curves of virgin aggregates and RAP after binder extraction.

Table 2
Main characteristics of the polymeric additive.

Aspect	Granules
Colour	Shades of grey
Apparent density at 25 °C	0.4 – 0.6 g/cm^3
Softening point	160–180 °C

selected compound does not meet this temperature criterion, its incorporation into the asphalt mixtures resulted in hybrid modification of the material. The manufacturer recommended a RPC dosage of 0.3 % by total weight of mixture; however, previous studies documented improvements in some mechanical properties for mixtures incorporating 0.5 % RPC [57].

The rejuvenating agent was a liquid bio-based product. Following the manufacturer's recommendation, it was used with a dosage of 0.3 % by weight of RAP weight, corresponding to approximately 6 % by weight of aged RAP binder. The rejuvenator was added directly to RAP and pre-blended for approximately 20 s, prior to mixing RAP with heated aggregate and filler, and subsequently with the bitumen. This procedure was adopted to promote the uniform distribution of the rejuvenator within the aged binder of RAP. The main properties of the rejuvenating product are indicated in Table 3.

The bituminous mixtures were characterized by a maximum nominal aggregate size of 16 mm (AC 16), commonly used for binder course applications. The target gradation and reference limits, established in accordance with [59], are illustrated in Fig. 2. The aggregate skeleton was obtained by combining 50 % RAP and 50 % of virgin aggregate plus filler. The mix design was based on existing type testing protocols issued by the mixing plant which provided aggregates for this experimental study. Therefore, further mix optimization including setting optimum bitumen content was not necessary since an approved real mix design was used.

Virgin aggregate was heated for approximately 4 h at 170 °C and then transferred to the laboratory mixer. About 20 min before adding the coarse aggregate, the RPC was evenly spread over the aggregate surface to allow preheating. Aggregates and RPC were then mixed for approximately 60 s before introducing the preheated RAP. The RAP material was heated for approximately 4 h at 130 °C, corresponding to the typical temperature used in asphalt plants equipped with a parallel drum. After the addition of RAP, the mixture of aggregates and RPC was mixed for 180 s, with the laboratory mixer temperature set equal to 160 °C.

Once all mineral components and RPC were homogenized, the bitumen and filler were added, and mixing continued for an additional 180 s. The total mixing sequence (180 + 180 s) followed the default preset on the laboratory mixer. For the wet-reference mixture containing SBS highly-modified binder, the total mixing time was the same (360 s), but aggregates were premixed without RPC. The remaining steps of the laboratory HMA production followed the same procedure. The compaction temperature was set at 150 °C for mixtures containing either Binder A or Binder B, and at 160 °C for the wet-modified mixture containing binder C. A total of six bituminous mixtures were produced and evaluated. Hybrid-modified mixtures included two mixtures containing 0.3 % RPC (RPC0.3 and RPC0.3 +RJ) and two mixtures containing 0.5 % RPC (RPC0.5 and RPC0.5 +RJ), with dosages expressed by total mixture weight. RPC0.3 and RPC0.5 were prepared with Binder A, while RPC0.3 +RJ and RPC0.5 +RJ combined Binder B with the rejuvenating agent. The remaining two mixtures were produced with Binder C (PMB, wet-modified mixture) and Binder A (NEAT, base control mixture without modification).

Table 3
Properties of the rejuvenating agent used in the investigation.

Aspect	Liquid
Colour	Brown - Purple
Density at 25 °C	0.90 ± 0.05 g/cm^3
Viscosity at 25 °C	100 ± 50 mPa s
Flash point	> 200 °C
Water content	< 2 %

The experimental work involved the preparation of 42 test specimens per mixture - comprising both beams and cylindrical samples - resulting in a total of 252 specimens. Beams were cut from larger rectangular slabs compacted using a roller compactor equipped with vertical sliding steel plates, following EN 12697–33 [64]. Cylindrical specimens were compacted using a standard Marshall impact compactor with 2 × 50 blows, following EN 12697–30 [65].

According to [59], the air voids content for AC 16 mixtures should range between 4 % and 6 % for mixtures designed for type testing. Therefore, the optimum binder content for each mixture was determined based on volumetric properties. Both NEAT and PMB met this requirement with a total binder content (TBC) of 4.55 % following the mix design in type testing protocol. The hybrid-modified mixtures required higher TBC values: RPC0.3 and RPC0.3 +RJ contained 4.85 %, while RPC0.5 and RPC0.5 +RJ contained 5.05 %. Notably, both the rejuvenator and RPC were included in the TBC calculation, based on the hypothesis that they function as binder extenders and contribute to the binding of the aggregate structure [55]. Furthermore, all mixtures were designed with comparable binders contents to ensure consistency across mixture groups; differences in mixtures composition are specified in Table 4.

3. Methods

3.1. Four-point bending tests

4PB tests for the characterization of the stiffness properties of the bituminous mixtures were carried out on prismatic asphalt beams at 0, 10, 20, and 30 °C, in accordance with EN 12697–26, Annex B [66]. Beam dimensions were 50.0 ± 1.0 mm in width, 52.5 ± 0.8 mm in height, and 405.0 ± 0.2 mm in length. A strain amplitude of 50 μm/m was applied for measurements, using a displacement-controlled sinusoidal waveform to maintain testing within the materials' linear viscoelastic range. Testing was performed across 11 loading frequencies: 0.1, 1.0, 2.0, 3.0, 5.0, 8.0, 10.0, 15.0, 20.0, 30.0, and 50.0 Hz. Although the standard recommends testing from the highest to the lowest temperature, prior experimental experience showed that beginning with 30 °C could lead to specimen damage. To mitigate this risk, the 30 °C tests were performed last. Five replicates were run per mixture and test temperature.

Stresses and strains recorded under sinusoidal loading were used to determine the norm of the complex modulus |E*| of mixtures. For the sake of simplicity and in line with the broader literature on viscoelastic characterization of asphalt materials, |E*| is hereinafter referred to as the dynamic modulus. The values of |E*| obtained at selected temperature and frequency were analysed for a direct assessment and comparison of various materials.

The collected data were also used for the construction of master curves at a reference temperature of 20 °C, following the procedure delineated in AGPT/T274 [67]. The following equation was considered to fit the experimental data:

$$\log_{10}|E^*| = \delta + \frac{\alpha}{1 + e^{\beta + \gamma \cdot \log_{10} f_r}} \tag{1}$$

where δ, α, β, γ are regression parameters, and f_r is the reduced frequency, computed as the product of the test frequency and the time-temperature shift factor a_T. This last one was modelled with the following expression:

$$\log_{10} a_T = a \cdot (T - T_{ref})^2 + b \cdot (T - T_{ref}) \tag{2}$$

where a and b are regression coefficients, and T and T_{ref} are the testing and reference temperatures, respectively.

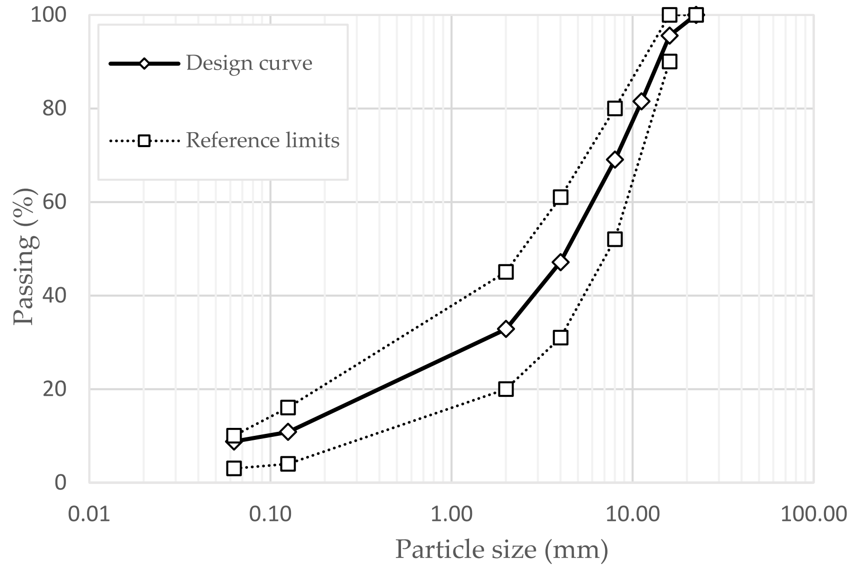


Fig. 2. Gradation curve and technical standard grading band.

Table 4
Composition of investigated mixtures.

Mix Code	Binder code	TBC (%)	RPC content (%)	Rejuvenator content (%)
NEAT	A	4.55	-	-
RPC0.3	A	4.85	0.3	-
RPC0.3 +RJ	B	4.85	0.3	0.15
PMB	C	4.55	-	-
RPC0.5	A	5.05	0.5	-
RPC0.5 +RJ	B	5.05	0.5	0.15

3.2. Wheel tracking tests

WT tests for the assessment of the rutting resistance of bituminous mixtures under repeated loading were carried out following EN 12697–22 [68]. Measurements were performed by means of a small-size device equipped with an air bath, at a temperature of 50 °C. Two asphalt slabs per mixture (each measuring 320 × 260 mm), pre-conditioned at target temperature for at least four hours, were used for testing. During WT test, a loaded steel wheel applied cyclic loading at a rate of 24 passes per minute, with rut depth continuously recorded. The test automatically terminated when either 10,000 load cycles were completed, or a maximum rut depth of 15 mm was reached. The rutting performance was evaluated based on three key parameters: the rut depth (RD), defined as the reduction in thickness of the test specimen caused by the repeated passes of the loaded wheel, the proportional rut depth (PRD), which represents the rut depth relative to the specimen height, and the wheel tracking slope (WTS), which quantifies the rate of rutting progression between 5000 and 10,000 cycles, as specified in [68].

3.3. Semi-circular bending tests

SCB tests for the assessment of crack propagation resistance and fracture behaviour of the bituminous mixtures were performed following a modified version of EN 12697–44 [69]. The procedure entailed using semi-circular Marshall-compacted specimens with a central notch [70]. The SCB half-disks had a height of 49.2 ± 1.9 mm and a thickness of 50.8 ± 0.9 mm, with the notch measuring 10 mm in length and 0.9 mm in width. The SCB tests were conducted at a fixed loading rate of 2.5 mm/min and at two test temperatures, equal to 0 °C and 15 °C. Six replicates were run for each temperature condition. Test data were used for the calculation of fracture parameters, including Fracture toughness (K_{Ic}) and Cracking Resistance Index (CRI), as expressed by the following equations:

$$K_{Ic} = \sigma_{max} \cdot Y_1 \cdot \sqrt{\pi \cdot a_i} \quad (3)$$

$$CRI = G_f / P_{max} \quad (4)$$

where σ_{max} is the maximum tensile stress at failure (MPa), Y_1 is the normalised stress intensity factor as defined in [69], a_i is the notch depth of i-th specimen (mm); G_f is the fracture energy, calculated as the ratio between the fracture work (the area underneath the load-displacement curve) and area of the ligament.

Each parameter provides insight into a specific aspect of fracture behaviour. K_{Ic} represents the maximum stress the mixture can resist before cracking; a higher K_{Ic} indicates better resistance to crack initiation. G_f captures the total energy required for crack propagation, including both pre- and post-peak behaviour. Such a parameter could be influenced by variations in peak load, thus Kaseer et al. [71] introduced CRI, which accounts for ductility properties by normalizing fracture energy with respect to peak load. A higher CRI value indicates more ductile failure, characterized by a smoother post-peak response.

3.4. Three-point bending tests

3PB tests were performed in accordance with [72] to evaluate the low-temperature flexural strength of bituminous mixtures. 3PB tests were carried out at a temperature of 0 °C, on beam specimens subjected to loading applied at a constant rate of 1.25 mm/min. During testing, both the applied load and the resulting deformation were continuously recorded up to the maximum load. From test data, the tensile flexural strength (R_i) was calculated using the following expression:

$$R_i = \frac{3}{2} \cdot \frac{P_{max} \cdot l}{b \cdot h^2} \quad (6)$$

where P_{max} is the maximum applied load (N), l is the support span (mm), and b and h are the width and height (mm) of the specimen, respectively. The fracture work (W_f), defined as the area under the load-displacement curve up to the peak load, was also calculated from experimental data using the same approach used for SCB test.

3.5. ANOVA, HSD, and Cohen's d analysis

A one-way Analysis of Variance (ANOVA) was performed to statistically evaluate the observed differences in the mechanical properties of the investigated bituminous mixtures, by assuming a significance level α = 0.05. According to the assumption, a statistically significant difference is indicated when the resulting p-value of ANOVA is less than 0.05. In such cases, the null hypothesis - stating that no differences exist among group means - is rejected. Conversely, if the p-value exceeds 0.05, the null hypothesis is retained, indicating no statistically significant differences among the compared mixtures. As required by ANOVA method, Levene's test was applied to evaluate the homogeneity of variances, and the Shapiro-Wilk test was used to verify the normality of the data distribution.

While ANOVA is effective in detecting the presence of at least one significant difference among group means, it does not identify which specific groups differ. Therefore, the Tukey Honestly Significant Difference (HSD) post hoc test was applied to determine the exact pairs of mixtures showing statistically significant differences. This complementary analysis enabled a more detailed interpretation of experimental results by performing multiple pairwise comparisons and highlighting the specific groups contributing to the overall variation observed in the dataset.

Such analyses were effective in identifying statistically significant differences between group means. However, they do not provide information about the magnitude of those differences. For such a reason, Cohen's d analysis was additionally performed to quantify the effect sizes between the investigated mixtures. All results of ANOVA, HSD, and Cohen's d analysis are reported in Appendix A.

4. Experimental results

4.1. Stiffness properties

Fig. 3 displays the average values of the dynamic modulus |E*| obtained from 4PB tests at different temperatures at the selected frequency of 5 Hz, assumed as reference. The error bars represent the standard deviations calculated from replicate measurements. The coefficients of variation (CoVs) were also calculated and reported in Appendix A (Table D1); they show that all mixtures exhibited values below 10 %, with just a few exceptions, thereby corroborating the reliability of the results.

It is firstly observed that the addition of the RPC to the mixtures led to a general increase in their stiffness. In fact, all modified mixtures exhibited higher |E*| values with respect to the base control mixture NEAT at all tested temperatures. The difference became more pronounced as the temperature increased: at 30 °C, NEAT showed stiffness values over 35 % lower than those of the other materials.

Increasing RPC content from 0.3 % to 0.5 % in hybrid-modified mixtures produced no statistically significant effects at 0 and 10 °C, as confirmed by HSD test values for pairs of mixtures sharing the same binder type: RPC0.3 vs RPC0.5 (Binder A) and RPC0.3 +RJ vs RPC0.5 +RJ (Binder B). Such findings were confirmed by Cohen's d (with only one exception corresponding to comparison between RPC0.3 +RJ and RPC0.5 +RJ at 10 °C), thus indicating that the hybrid modification method became increasingly effective with the increase of temperature to an extent that depended on RPC content.

The stiffening behaviour of RPC-modified mixtures can be mechanistically attributed to the presence of dispersed polymer particles partially embedded within the mastic phase, which increase internal friction and restrict binder flow around the aggregate framework. This produces a composite-like structure where the polymer inclusions act as rigid fillers, enhancing the elastic response of the material without complete polymer dissolution. At elevated temperatures, the softened binder allowed better wetting and dispersion of polymer particles, amplifying this reinforcing effect; at low temperatures, binder phase of the mixture tended towards the glassy behaviour with dominance of the aged RAP binder and its reduced molecular mobility, that suppressed the contribution of the RPC.

It is also observed that the combined use of Binder B with the rejuvenating agent led to lower mean values of stiffness compared to Binder A. This clearly emerges from the comparison of mixtures having the same polymer dosage (RPC0.3 vs RPC0.3 +RJ and RPC0.5 vs RPC0.5 +RJ) at almost all temperatures. Exceptions were observed at 20 and 30 °C, where RPC0.5 +RJ outperformed RPC0.5. Such differences were corroborated by Cohen's d, which indicated moderate and high effect sizes at 20°C and 30 °C, respectively. Nevertheless, output of HSD tests provided p-values generally higher than 0.05 for the above-mentioned pairwise comparisons, highlighting the need for further experimental data for better understanding this specific aspect. It can be argued that the rejuvenating agent acted as a softener of Binder B blended with the aged binder of RAP. However, as the binder phase softened, the influence of the polymer became progressively more dominant on the overall performance of the investigated mixtures. The reference wet-modified mixture PMB exhibited intermediate properties compared to the hybrid ones.

Further insights can be obtained from the analysis of TS index, calculated as the ratio between the mean stiffness moduli at 0 °C and at 30 °C. Such a parameter was introduced to enable a straightforward evaluation of temperature sensitivity based directly on stiffness measurements, rather than other parameters derived from rheological modelling outputs. Lower TS values reflect reduced sensitivity to temperature variations and improved thermal stability of the mixture. Fig. 3 shows that RPC0.5 and RPC0.5 +RJ (both containing the same RPC content equal to 0.5 %) yielded the lowest TS values, while control

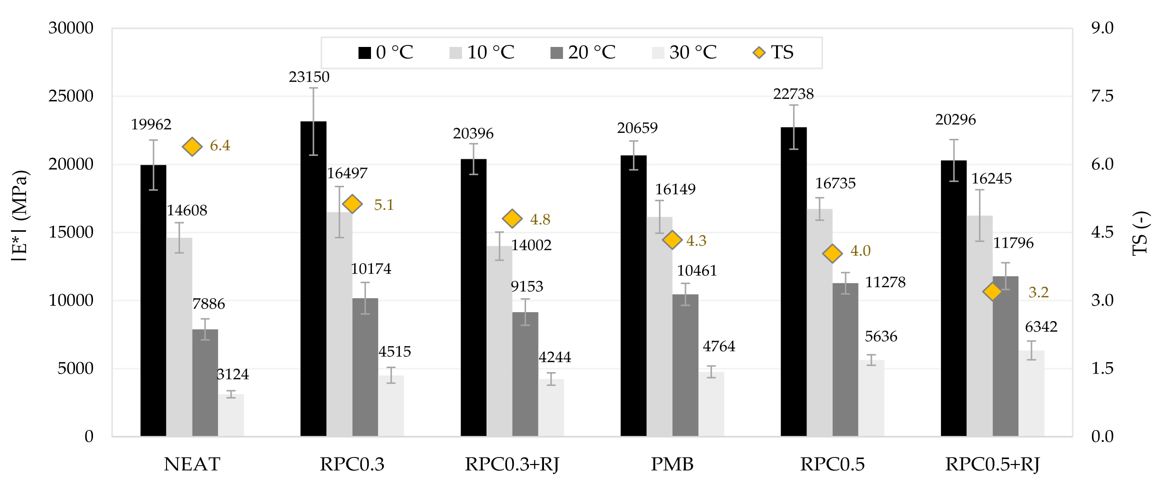


Fig. 3. |E*| of mixtures at 0, 10, 20, and 30 °C with corresponding TS values determined from 4PB tests at 5 Hz.

NEAT with no modifiers recorded the highest one. Such a finding confirms the beneficial effects of RPC on the thermal stability of hybrid mixtures. This reduced temperature susceptibility suggests that RPC improves the balance of viscoelastic responses in the mastic phase not only by physically constraining binder movement, but also by creating a dispersed, semi-rigid particle network that simultaneously promotes mechanical interlocking and increases the mastic's effective viscosity. These combined actions limit thermal softening and mitigate binder fluidity at elevated temperatures. The reduced gap between |E*| values associated with reduced TS values in RPC0.5 and RPC0.5 +RJ further highlights the impact on stiffness properties of higher RPC dosage at higher temperatures. Again, PMB showed intermediate performance compared to the hybrid mixtures containing RCP.

Fig. 4 shows the master curves derived from the 4PB test data, together with the fitting parameters (δ, α, β, and γ) used for their construction. The curves describe the variation of the dynamic modulus as a function of reduced frequency at a fixed reference temperature (assumed equal to 20 °C), thus complementing the analysis on stiffness properties of mixtures and their variation with loading time.

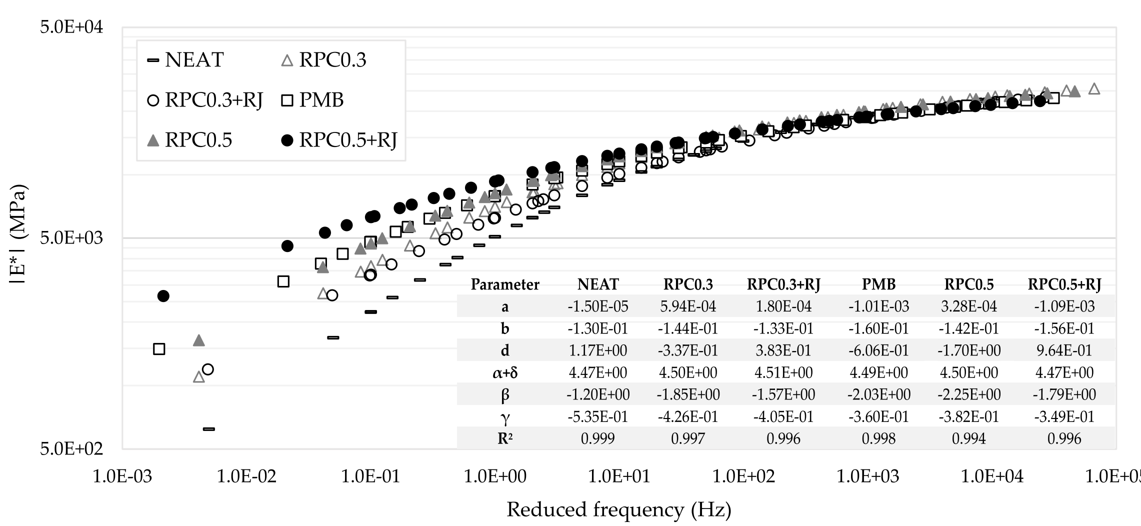
It is observed that in the higher frequency domain (lowest temperatures), master curves tend to overlap with each other, indicating a progressively reduced influence of the RPC on mixtures response. Since the sum δ+ α represents the upper asymptote of the sigmoidal function, their similar values confirm that differences among mixtures are minimal. Conversely, the influences of the polymer and its dosage become progressively relevant in the intermediate and low frequency ranges, reinforcing the assumption that low temperatures act as a "freezer" for the polymer influence. All modified mixtures (both via wet and hybrid methods) exhibited significantly higher stiffness compared to the base control NEAT without any modification. Moreover, RPC0.5 and RPC0.5 +RJ showed higher |E*| values compared to RPC0.3 and RPC0.3 +RJ, respectively, confirming that the beneficial effect of the polymer is more pronounced at higher temperatures, where the rest of the binder phase is softer. Notably, the reference wet-modified mixture PMB is characterized by intermediate stiffness properties with respect to the hybrid mixtures. The stiffness response is governed by the temperature: at low temperatures, wet modification tends to soften the binder phase with respect to the hybrid modification counterpart. However, such an outcome is not necessarily a disadvantage: lower stiffness may enhance ductility and cracking resistance performance. For such a reason, other tests must be investigated. As the temperature increases, the hybrid-modification exhibits a stronger influence: at intermediate temperatures, similar |E*| values were observed for RPC0.3, PMB, and RPC0.5; at higher temperatures, mixtures with increased RPC content exhibited a marked stiffness gain. Moreover, it is worth noting that mixture PMB contained approximately 6 % SBS polymer by bitumen weight, a proportion comparable to the polymer content in RPC0.3 (~6.5 % by bitumen weight) but significantly lower than in RPC0.5 (~11 % by bitumen weight), explaining the stronger stiffness response of higher RPC dosages at elevated temperatures.

From the comparison of RPC0.5 and RPC0.5 +RJ at intermediate and low frequency domains, it is observed that the combined use of Binder B with rejuvenating agent resulted in a higher rate of stiffness with respect to Binder A when the higher polymer content was added to the mixture. Overall, RPC0.5 +RJ yielded the highest stiffness levels compared to other mixtures at the intermediate and low frequencies, confirming that in this range the polymer stiffening effect is predominant over the softening action of the rejuvenator. Conversely, at higher frequencies, the rejuvenator reduced stiffness more effectively.

Mixture NEAT exhibited the highest β value and lowest γ values, indicating that its master curve is more shifted to the right and characterized by a sharper shape. It is also observed that β values decreased and γ values increased with RPC content (RPC0.3 vs RPC0.5, RPC0.3 +RJ vs RPC0.5 +RJ). Moreover, similar δ+ α values across mixtures led to a perfectly reversed ranking order between β and γ, highlighting the interplay between position and steepness of the master curve profiles. From a mechanistic standpoint, the evolution of the master curves with polymer dosage and binder type can be explained as follows. At low temperatures (high frequencies), the composite stiffness is governed by the rigid RAP binder and the mineral skeleton, resulting in overlapping curves. As the loading time increases or temperature rises, the dispersed RPC particles begin to interact more effectively with the softened binder, constraining molecular mobility and delaying the viscous response. This yields flatter, less temperature-sensitive master curves and explains the higher |E*| observed at low frequencies. The decreasing β and increasing γ parameters with polymer addition confirm this transition from a brittle (rapid-relaxation system) to a more elastic (energy-storing composite) structure typical of physically reinforced hybrid mixtures. The outcomes emerged from the analysis of master curves reported in Fig. 4 are fully coherent with those arose from the analysis of data reported in Fig. 3, confirming a full correspondence between time and temperature effects in the time-temperature window investigated in the study.

4.2. Rutting performance

Fig. 5 presents the results obtained from WT tests, expressed in terms of RD, PRD, and WTS parameters. The diagram reports the average



Parameter	NEAT	RPC0.3	RPC0.3+RJ	PMB	RPC0.5	RPC0.5+RJ
a	-1.50E-05	5.94E-04	1.80E-04	-1.01E-03	3.28E-04	-1.09E-03
b	-1.30E-01	-1.44E-01	-1.33E-01	-1.60E-01	-1.42E-01	-1.56E-01
d	1.17E+00	-3.37E-01	3.83E-01	-6.06E-01	-1.70E+00	9.64E-01
α+δ	4.47E+00	4.50E+00	4.51E+00	4.49E+00	4.50E+00	4.47E+00
β	-1.20E+00	-1.85E+00	-1.57E+00	-2.03E+00	-2.25E+00	-1.79E+00
γ	-5.35E-01	-4.26E-01	-4.05E-01	-3.60E-01	-3.82E-01	-3.49E-01
R^2	0.999	0.997	0.996	0.998	0.994	0.996

Fig. 4. |E*| vs. reduced frequency master curves at reference temperature of 20°C.

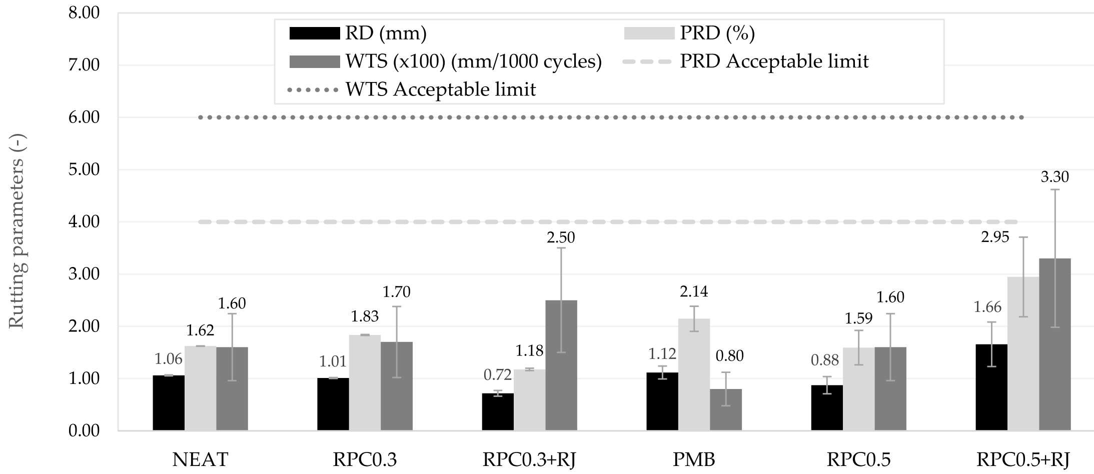


Fig. 5. Mean values of RD, PRD, and WTS (×100) rutting performance parameters obtained from WT tests at 50 °C.

values and error bars corresponding to the range between maximum and minimum recorded values. Fig. 5 also reports the specification thresholds (depicted with dashed lines in the graph) defined by [59], corresponding to PRD < 4 % and WTS < 0.06 mm/1000 cycles determined after 10,000 cycles. Due to the limited number of test replicates as required by [68], statistical tests could not be performed.

It is observed that all bituminous mixtures complied with the specification limits, indicating adequate rutting resistance under elevated temperature and repeated loading conditions.

The various mixtures reached very similar final rut depths RD at the end of tests, with values around 1 mm after 10,000 loading cycles. Consequently, mixtures with higher PRD values generally showed lower WTS, and vice versa.

No clear trends were observed regarding modifier type or dosage. It is then argued that the rutting performance of the considered mixtures is mostly governed by RAP, with limited impact of modification processes. In fact, the high amount of aged and stiff binder in RAP-rich mixtures prevailed over polymer effects in improving resistance to permanent deformation of materials. Mechanistically, this can be explained by the dominance of the stiff, highly viscous aged binder contained in RAP, which constrains shear deformation under load. In hybrid mixtures, the polymeric modifier primarily provides physical reinforcement through particle inclusion within the mastic rather than altering binder rheology. Because its effect is localized and not chemically integrated into the binder phase, the overall contribution to rutting resistance remains limited in RAP-rich systems.

A specific focus deserves to be made on RPC0.5 +RJ, which manifested the highest values for all considered parameters. This appears quite surprising, since in contrast to previous results gathered from stiffness tests. Such a response may indicate potential problems of excessive softening of the binder matrix caused by the rejuvenator added at a higher TBC content when the temperature rises above a certain level. In fact, the rejuvenator might have reduced binder viscosity, which can weaken interfacial adhesion between the polymer particles and the surrounding mastic, promoting localized flow and reduced shear resistance. However, such a result is not coherent with existing literature [40]. Moreover, it is worth noting that values of parameters are well below the specification limits and vary in quite narrow ranges. Therefore, it may be concluded that the observed anomalies of RPC0.5 +RJ can be attributed to experimental variability associated with specimen preparation and laboratory test procedures. To further support this explanation, it is observed that the error bars reported in Fig. 5 display notably wider ranges for RPC0.5 +RJ compared to the other mixtures. Given the limited number of tests, the performance of this mixture type may have been influenced by a single laboratory slab.

4.3. Fracture toughness and cracking resistance

Figs. 6 and 7 report the results of SCB tests carried out on mixtures. Both diagrams display the mean values of the considered parameters, with error bars representing the corresponding standard deviations. CoVs are reported in Appendix A (Table D1) indicating average values of around 12.

As observed from the analysis of K_{Ic} parameter at 0°C (Fig. 6), higher RPC dosages improved the resistance to crack initiation, with RPC0.5 exhibiting the highest value, strictly followed by RPC0.5 +RJ. The differences between these two mixtures were minimal, as confirmed by HSD and Cohen's d analyses. Moreover, results suggest that hybrid-modified mixtures incorporating the softer binder exhibited slightly superior resistance compared to their counterparts produced with the harder binder and the rejuvenating agent (RPC0.3 vs RPC0.3 +RJ and RPC0.5 vs RPC0.5 +RJ). The base control NEAT showed intermediate responses. However, the comparison among NEAT, RPC0.3, RPC0.3 +RJ, and PMB revealed neither statistical differences nor large Cohen's d values, confirming the prominent role of RAP on the resistance to crack initiation at low temperatures. Such limited differences are consistent with findings reported in the existing literature [73].

At 15 °C, all modified mixtures outperformed NEAT, with PMB achieving the highest k_{IC} value. At this temperature, the mixture RPC0.5 +RJ combining Binder B and the rejuvenator slightly outperformed the mixture with the softer Binder A, reversing the trend observed at lower temperatures. Such an outcome was once again supported by Cohen's d, which indicated a moderate effect size despite a higher p-value, likely due to a limited number of test replicates. This temperature-dependent behaviour can be mechanistically explained by the change in fracture mode from brittle to ductile. At low temperatures, the response is dominated by the stiff RAP binder, which limits strain accommodation and causes brittle failure regardless of polymer addition. As temperature increases, the dispersed RPC particles act as local stress relievers and crack-bridging inclusions, improving ductility and energy dissipation during fracture propagation. The rejuvenator enhances binder diffusion and reduces stiffness gradients at RAP–virgin bitumen interfaces, facilitating more uniform stress transfer and delaying crack initiation. In other words, being K_{Ic} the maximum stress the mixture can resist before cracking, at low temperature the slightly higher stiffness provided by higher RPC contents provides only a marginal improvement. In this condition, ductility and energy dissipation are largely "frozen" by the presence of RAP, highlighting no significant differences. At intermediate temperatures, the effect of hybrid modification becomes more evident: the softening of the binder phase and the improved dispersion of the polymer within the matrix enhance material ductility. However, this also results in a sharp reduction in K_{Ic} compared

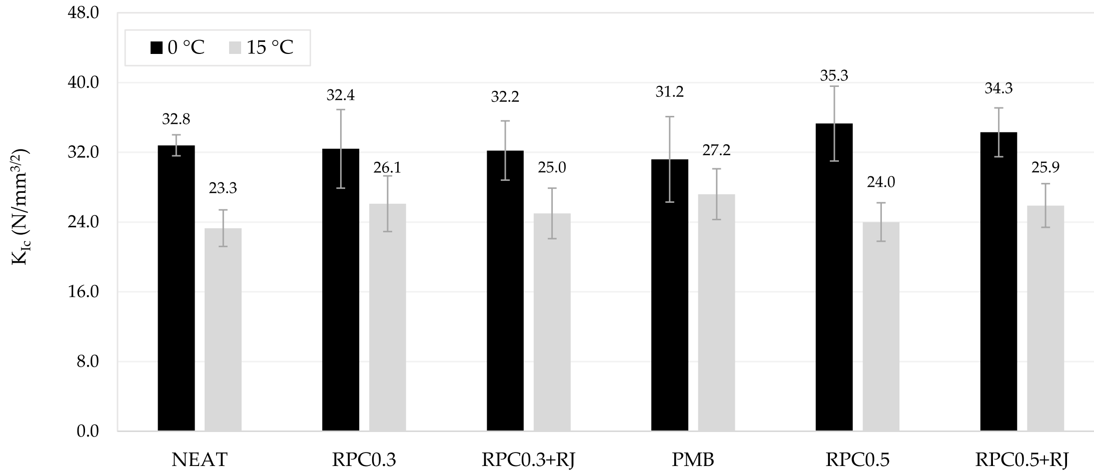


Fig. 6. Fracture toughness (K_{Ic}) of asphalt mixtures at 0 °C and 15 °C.

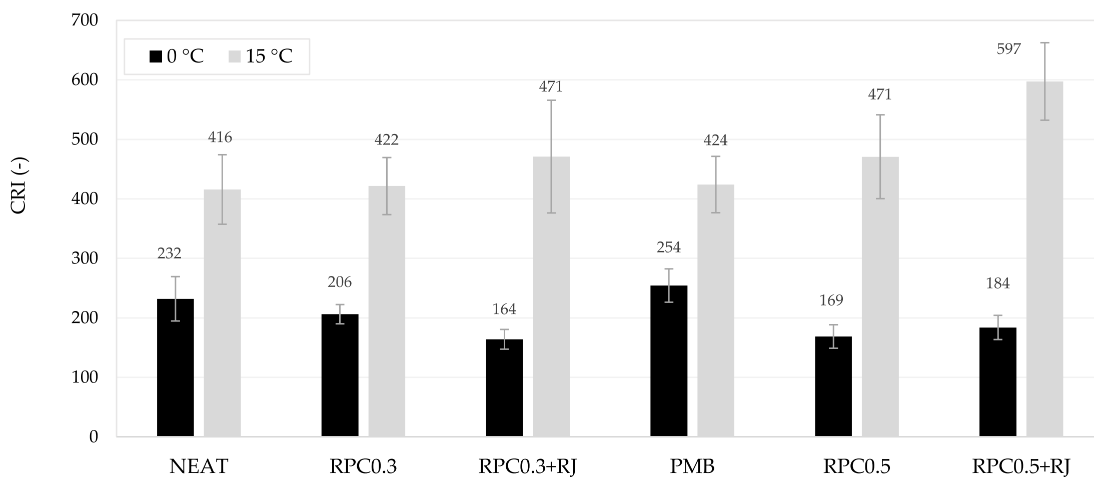


Fig. 7. Cracking Resistance Index (CRI) of asphalt mixtures at 0 °C and 15 °C.

to wet modified mixture PMB.

The results of CRI parameter (Fig. 7) show that at 0 °C the reference wet-modified PMB manifested the highest CRI values, followed by base control mixtures NEAT. Overall, the hybrid-modified mixtures required less energy for crack propagation compared to both unmodified and wet-modified HMAs, as demonstrated by HSD and Cohen's d analyses. No consistent trends were observed concerning polymer dosage or binder type in hybrid-modified mixtures. The observed differences were generally limited, as supported by HSD results and partially by Cohen's d, which identified negligible to medium effect sizes.

Different outcomes emerged at 15 °C. An improvement in CRI was observed with the addition of RPC; in fact, all hybrid-modified mixtures outperformed NEAT and PMB (except the case of RPC0.3 which was nearly identical to PMB according to HSD and Cohen's results). Moreover, the increase in RPC content yielded increased CRI values. These results suggest that softening of the binder phase caused by the raise in temperature allowed the ductile properties of polymer to be emphasized, with consequent ductile failure of corresponding mixtures.

This indicates that the ductile characteristics of the dispersed plastic phase become more effective when the binder matrix softens, allowing the particles to deform and redistribute stresses rather than acting as rigid inclusions. In similar terms, the effect of the rejuvenator is relevant at intermediate temperatures promoting a better interaction between virgin and aged RAP binders, thus reducing interfacial stress concentrations and leading to improved crack-propagation resistance.

The highest level of CRI was achieved by RPC0.5 +RJ, which exceeded NEAT by over 30 %. It is also noteworthy that mixtures RPC0.3 +RJ and RPC0.5 +RJ outperformed RPC0.3 and RPC0.5, respectively, as demonstrated by Cohen's d values. Such outcomes suggest a positive synergistic effect of combining the rejuvenating agent with the harder Binder B.

4.4. *Flexural strength and fracture work*

Figure 8 shows the mean values of flexural strength (R_i) and fracture work (W_f) obtained from 3PB tests at 0 °C, with error bars indicating the corresponding standard deviations. A total of four replicates were tested for each investigated mixture. As reported in Table D1 (Appendix A), all mixtures exhibited acceptable CoV values, with just a few cases slightly exceeding 10 %. The highest CoV was observed for the W_f of RPC0.3 (approximately 17 %), which can likely be attributed to its relatively low mean value. According to the definitions, a higher R_i indicates a better cracking resistance, as well as a higher W_f reflects a greater energy absorption capacity before failure. All modified mixtures exhibited higher strength compared to base control mixture NEAT, except for the average RPC0.3 which showed the lowest R_i value overall. Although, the differences between RPC0.3 and NEAT were not statistically significant, Cohen's d values confirmed a moderate effect size. This confirms the limited contribution of lower RPC contents without rejuvenating agents due to the high RAP content.

The highest R_i values were recorded in RPC0.3 +RJ and RPC0.5 +RJ. Results of 4PB tests shown in Figs. 3 and 4 indicated lower

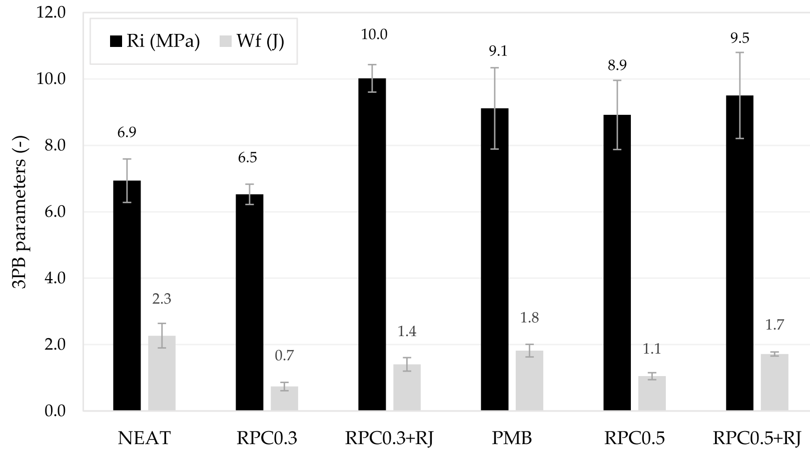


Figure. 8. Flexural strength and fracture work values of asphalt mixtures from 3PB tests.

values of |E*| for these two mixtures compared to the other two hybrid ones; therefore, the reduced stiffness consequent to the use of stiffer Binder B combined with rejuvenating agent reflected on an increased flexural strength. Moreover, the rejuvenator contributed to improved blending and partial reactivation of the aged RAP binder, further enhancing the overall flexural strength of the mixtures. PMB manifested again intermediate performance, lying between the hybrid mixtures prepared with Binder A (RPC0.3 and RPC0.5) and those produced with Binder B and the rejuvenator (RPC0.3 +RJ and RPC0.5 +RJ).

The influence of rejuvenator combined with the harder binder also emerged from the analysis of fracture work W_f, which measures the energy required to propagate a crack. Findings indicated a 100 % increase in W_i for RPC0.3 +RJ compared to RPC0.3, and a 54 % increase for RPC0.5 +RJ with respect to RPC0.5, validating that an improved RAP binder reactivation and higher RPC contents can help hybrid-modified mixtures achieving properties comparable to those of the wet-modified reference mixture. Such an outcome corroborates that at low temperatures, the excessively high |E*| of hybrid-modified mixtures without the rejuvenator combined with the limited effectiveness of the hybrid modification method, results in mixtures with brittle characteristics. Consistent with the SCB test results, which highlighted higher CRI values in PMB, W_f values from the 3PB tests confirmed that the SBS highly-modified binder mixture can dissipate more energy before failure. This characteristic may be attributed to the binder phase of the wet modification method: at lower temperatures the bitumen is inherently stiffer, and the homogeneous dispersion of the polymer inside the bitumen phase provides higher levels of ductility with respect to the hybrid modification counterpart.

5. Summary and conclusions

The experimental study investigated the combined effect of an RPC and high RAP content (50 %) on the mechanical properties of bituminous mixtures under different test conditions. The use of a rejuvenating agent in combination with a harder neat binder in comparison with the use of a sole softer neat binder was also investigated. A total of six asphalt mixtures, including two reference mixtures produced without RPC and with a SBS highly-modified binder, were evaluated and compared through an extensive laboratory experimentation. Testing program entailed determination of stiffness properties, rutting performances, fracture and crack propagation resistance at low and intermediate temperatures, and flexural strength of the mixtures.

Overall, the experimental findings demonstrated that the combined use of recycled plastic and high content of RAP represents a viable and promising solution for the production of sustainable asphalt mixtures. When employed together with a suitably selected neat binder and a rejuvenating agent, the modification with recycled plastics may deliver performance levels comparable or even exceeding those of mixtures incorporating a SBS highly-modified binder, especially at intermediate and high temperatures.

Specific conclusions from the study can be drawn as follows:

- Stiffness properties of investigated mixtures at lower temperatures were mainly governed by the high RAP content, which overwhelmed the contribution of RPC. At intermediate and high temperatures, the influence of RPC added via hybrid method became more effective, promoting polymer dispersion and amplifying the reinforcing effect, which consequent enhancement of the overall stiffness of materials.
- Rutting performances of bituminous mixtures complied with specification limits assumed as reference, thus showing adequate resistance to permanent deformation under elevated temperatures and repeated loading. No significant differences were observed among various mixture groups, indicating a prevalence of the effects of RAP on mixture response.
- Fracture properties and cracking resistance of mixtures were influenced by RPC to an extent that depended on test temperature. At lower temperature, the hybrid modification was partially hindered by the stiffness of the binder phase: this slightly enhanced the maximum stress the mixture can resist before cracking, while affecting ductility properties. At intermediate temperature, fracture properties were improved with the addition of RPC. The increased RPC content led to higher CRI values, with differences exceeding 30 % compared to the base control mixture containing neat binder without any modification.
- Hybrid mixtures modified with RPC generally exhibited higher flexural strengths compared to base control mixture. Wet mixture containing SBS highly-polymer modified binder showed intermediate strength properties between the hybrid mixtures prepared with softer binder and those produced with harder binder combined with the rejuvenator. When referring to fracture work, wet mixtures exhibited higher values compared to hybrid ones revealing the ability to dissipate more energy before failure.
- The bio-based rejuvenator improved blending and partial reactivation of the aged RAP binder. Its combined use with the harder bitumen led to hybrid mixtures that outperformed the hybrid mixtures containing the sole softer bitumen, in terms of cracking resistance, flexural strength and fracture work. Results indicated 100 % and 54 % increases in W_i values consequent to use of rejuvenator at lower and higher RPC dosages, respectively.

Further experimentation is needed to support the conclusions of the paper. The array of materials should be expanded to include recycled plastics and rejuvenating agents of different types and origins. The behaviour of the mixtures in terms of moisture damage should also be included in the experimental work. Moreover, laboratory findings should be validated through field testing data and complemented by environmental and economic evaluations, such as life-cycle assessment and life-cycle cost analysis.

CRediT authorship contribution statement

Jan Valentin: Writing – review & editing, Supervision, Resources, Methodology. **Davide Dalmazzo:** Writing – review & editing, Methodology, Formal analysis. **Amira Ben Ameur:** Writing – original draft, Investigation, Data curation. **Joseph Nicolas La Macchia:** Writing – original draft, Investigation, Formal analysis, Conceptualization. **Orazio Baglieri:** Writing – review & editing, Supervision, Methodology, Funding acquisition, Conceptualization.

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Declaration of Competing Interest

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

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Appendix A

This appendix includes all the outputs of statistical analyses performed on test data. Tables A1-A3 report the p-values obtained from one-way ANOVA, along with results of the Shapiro–Wilk (S-W) and Levene (L) tests used to verify the assumptions required for ANOVA. Tables B1-B10 present the p-values obtained from HSD tests, to identify statistically significant differences between pairs of investigated mixtures. Tables C1-C10 illustrate Cohen's d: an estimate of the magnitude of an effect independent of sample size. Finally, Table D1 lists all CoV values.

Table A1
P-values from L, S-W, and ANOVA tests of the |E*| parameters at 5 Hz (4PB test)

Parameter	P-value		
	Levene	Shapiro-Wilk	ANOVA
|E*| (0 °C)	0.704	0.419	0.031
|E*| (10 °C)	0.217	0.624	0.019
|E*| (20 °C)	0.944	0.231	< 0.001
|E*| (30 °C)	0.527	0.853	< 0.001

Table B1
P-values from post-hoc HSD tests of the |E*| parameters at 5 Hz at 0 °C (4PB test)

HSD	P-value					
	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	0.084	0.999	0.984	0.128	> 0.999
RPC0.3		-	0.218	0.261	0.999	0.180
RPC0.3 +RJ			-	> 0.999	0.321	> 0.999
PMB				-	0.383	> 0.999
RPC0.5					-	0.280
RPC0.5 +RJ						-

Table C1
Effect sizes of the |E*| parameters at 5 Hz at 0 °C (4PB test)

Cohen's d	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	-1.5	-0.3	-0.5	-1.6	-0.2
RPC0.3		-	1.4	1.4	0.2	1.4
RPC0.3 +RJ			-	-0.2	-1.6	0.1
PMB				-	-1.5	0.3
RPC0.5					-	1.5
RPC0.5 +RJ						-

Table B2
P-values from post-hoc HSD tests of the |E*| parameters at 5 Hz at 10 °C (4PB test)

HSD	P-value					
	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	0.316	0.977	0.467	0.159	0.466
RPC0.3		-	0.096	0.999	> 0.999	> 0.999
RPC0.3 +RJ			-	0.152	0.038	0.163
PMB				-	0.980	> 0.999
RPC0.5					-	0.993
RPC0.5 +RJ						-

Table C2
Effect sizes of the |E*| parameters at 5 Hz at 10 °C (4PB test)

Cohen's d	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	-1.3	0.6	-1.3	-2.2	-1.1
RPC0.3		-	1.7	0.2	-0.2	0.1
RPC0.3 +RJ			-	-1.9	-2.9	-1.5
PMB				-	-0.6	-0.1
RPC0.5					-	0.4
RPC0.5 +RJ						-

Table B3
P-values from post-hoc HSD tests of the |E*| parameters at 5 Hz at 20 °C (4PB test)

HSD	P-value					
	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	0.012	0.274	0.002	< 0.001	< 0.001
RPC0.3		-	0.559	0.997	0.478	0.159
RPC0.3 +RJ			-	0.244	0.014	0.003
PMB				-	0.713	0.281
RPC0.5					-	0.954
RPC0.5 +RJ						-

Table C3
Effect sizes of the |E*| parameters at 5 Hz at 20 °C (4PB test)

Cohen's d	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	-2.4	-1.4	-3.2	-4.4	-4.5
RPC0.3		-	1.0	-0.3	-1.1	-1.5
RPC0.3 +RJ			-	-1.5	-2.4	-2.7
PMB				-	-1.0	-1.5
RPC0.5					-	-0.6
RPC0.5 +RJ						-

Table B4
P-values from post-hoc HSD tests of the |E*| parameters at 5 Hz at 30 °C (4PB test)

HSD	P-value					
	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	0.003	0.012	< 0.001	< 0.001	< 0.001
RPC0.3		-	0.953	0.967	0.020	< 0.001
RPC0.3 +RJ			-	0.522	< 0.001	< 0.001
PMB				-	0.074	< 0.001
RPC0.5					-	0.266
RPC0.5 +RJ						-

Table C4
Effect sizes of the |E*| parameters at 5 Hz at 30 °C (4PB test)

Cohen's d	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	-3.2	-3.0	-4.6	-7.6	-6.5
RPC0.3		-	0.5	-0.5	-2.3	-2.8
RPC0.3 +RJ			-	-1.2	-3.3	-3.7
PMB				-	-2.1	-2.8
RPC0.5					-	-1.3
RPC0.5 +RJ						-

Table A2
P-values from L, S-W, and ANOVA tests of the parameters derived from SCB tests

Parameter	P-value		
	Levene	Shapiro-Wilk	ANOVA
K_{Ic} (0 °C)	0.156	0.778	0.564
CRI (0 °C)	0.113	0.455	< 0.001
Log K_{Ic} (15 °C)*	0.873	0.092	0.222
CRI (15 °C)	0.207	0.136	0.006

* NOTE: a data transformation was required to satisfy the assumptions of normality. Although the Shapiro-Wilk test returned a p-value close to 0.05, it fell slightly below the threshold, indicating a deviation from normal distribution. As a consequence, a logarithmic transformation of the dataset was applied to normalize the data prior to ANOVA investigation.

Table B5
P-values from post-hoc HSD tests of K_{Ic} at 0 °C (SCB test)

HSD	P-value					
	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	> 0.999	> 0.999	0.990	0.903	0.990
RPC0.3		-	> 0.999	0.997	0.807	0.964
RPC0.3 +RJ			-	0.998	0.781	0.954
PMB				-	0.530	0.796
RPC0.5					-	0.998
RPC0.5 +RJ						-

Table C5
Effect sizes of K_{Ic} at 0 °C (SCB test)

Cohen's d	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	0.1	0.2	0.4	-0.8	-0.7
RPC0.3		-	0.1	0.3	-0.7	-0.5
RPC0.3 +RJ			-	0.2	-0.8	-0.7
PMB				-	-0.9	-0.8
RPC0.5					-	0.3
RPC0.5 +RJ						-

Table B6
P-values from post-hoc HSD tests of CRI at 0 °C (SCB test)

HSD	P-value					
	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	0.799	0.010	0.835	0.088	0.029
RPC0.3		-	0.336	0.254	0.862	0.507
RPC0.3 +RJ			-	0.001	0.840	> 0.999
PMB				-	0.009	0.003
RPC0.5					-	0.955
RPC0.5 +RJ						-

Table C6
Effect sizes of CRI at 0 °C (SCB test)

Cohen's d	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	0.8	2.4	-0.7	2.0	1.7
RPC0.3		-	2.6	-2.0	2.0	1.2
RPC0.3 +RJ			-	-4.1	-0.3	-1.1
PMB				-	3.5	3.0
RPC0.5					-	-0.8
RPC0.5 +RJ						-

Table B7
P-values from post-hoc HSD tests of Log K_{Ic} at 15 °C (SCB test)

HSD	P-value					
	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	0.585	0.909	0.219	0.998	0.612
RPC0.3		-	0.986	0.978	0.809	> 0.999
RPC0.3 +RJ			-	0.746	0.990	0.990
PMB				-	0.383	0.971
RPC0.5					-	0.831
RPC0.5 +RJ						-

Table C7
Effect sizes of K_{Ic} at 15 °C (SCB test)

Cohen's d	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	-1.0	-0.7	-1.5	-0.3	-1.1
RPC0.3		-	0.4	-0.4	0.8	0.1
RPC0.3 +RJ			-	-0.8	0.4	-0.3
PMB				-	1.2	0.5
RPC0.5					-	-0.8
RPC0.5 +RJ						-

Table B8
P-values from post-hoc HSD tests of CRI at 15 °C (SCB test)

HSD	P-value					
	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	> 0.999	0.899	> 0.999	0.880	0.013
RPC0.3		-	0.952	> 0.999	0.943	0.033
RPC0.3 +RJ			-	0.934	> 0.999	0.136
PMB				-	0.918	0.011
RPC0.5					-	0.098
RPC0.5 +RJ						-

Table C8
Effect sizes of CRI at 0 °C (SCB test)

Cohen's d	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	-0.1	-0.7	-0.2	-0.8	-2.9
RPC0.3		-	-0.6	-0.1	-0.8	-2.9
RPC0.3 +RJ			-	0.7	0.0	-1.6
PMB				-	-0.8	-3.0
RPC0.5					-	-1.9
RPC0.5 +RJ						-

Table A3
P-values from L, S-W, and ANOVA tests of the parameters derived from 3PB tests

Parameter	P-value		
	Levene	Shapiro-Wilk	ANOVA
R_i	0.072	0.792	0.003
W_f	0.204	0.925	< 0.001

Table B9
P-values from post-hoc HSD tests of R_i at 0 °C (3PB test)

HSD	P-value					
	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	0.996	0.022	0.118	0.182	0.043
RPC0.3		-	0.014	0.070	0.107	0.027
RPC0.3 +RJ			-	0.871	0.757	0.989
PMB				-	> 0.999	0.993
RPC0.5					-	0.963
RPC0.5 +RJ						-

Table C9
Effect sizes of R_i at 0 °C (3PB test)

Cohen's d	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	0.7	-5.5	-2.3	-2.3	-2.5
RPC0.3		-	-9.7	-2.7	-2.9	-2.9
RPC0.3 +RJ			-	0.9	1.3	0.5
PMB				-	0.2	-0.3
RPC0.5					-	-0.5
RPC0.5 +RJ						-

Table B10
P-values from post-hoc HSD tests of W_f at 0 °C (3PB test)

HSD	P-value					
	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	< 0.001	0.010	0.334	< 0.001	0.178
RPC0.3		-	0.042	0.001	0.548	0.002
RPC0.3 +RJ			-	0.342	0.420	0.571
PMB				-	0.011	0.998
RPC0.5					-	0.024
RPC0.5 +RJ						-

Table C10
Effect sizes of W_f at 0 °C (3PB test)

Cohen's d	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
NEAT	-	5.6	2.9	1.5	4.9	2.1
RPC0.3		-	-3.9	-6.7	-2.7	-9.8
RPC0.3 +RJ			-	-2.1	2.3	-2.1
PMB				-	5.3	0.7
RPC0.5					-	-7.5
RPC0.5 +RJ						-

Table 11
Coefficient of Variations (CoV) from all the investigated tests

CoV (%)	NEAT	RPC0.3	RPC0.3 +RJ	PMB	RPC0.5	RPC0.5 +RJ
$\|E^*\|$ (0 °C)	9.2	10.7	5.5	5.1	7.1	7.6
$\|E^*\|$ (10 °C)	7.6	11.4	7.4	7.4	4.9	11.7
$\|E^*\|$ (20 °C)	9.8	11.4	10.5	7.8	6.9	8.3
$\|E^*\|$ (30 °C)	8.4	12.9	10.7	9.0	6.9	10.9
K_{Ic} (0 °C)	3.7	13.9	10.6	15.7	12.2	8.2
CRI (0 °C)	16.1	7.8	10.1	11.1	11.7	11.1
K_{Ic} (15 °C)	9.0	12.3	11.6	10.7	9.2	9.7
CRI (15 °C)	14.1	11.4	20.1	11.2	15.0	10.9
R_i (0 °C)	9.4	4.7	4.1	13.4	11.7	13.6
W_f (0 °C)	16.3	17.4	14.6	10.5	10.0	3.5

Data availability

Data will be made available on request.

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