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ENVIRONMENTAL AND FUNCTIONAL ROLE OF RECYCLED CRUMB RUBBER IN HOT-APPLIED JOINT SEALANTS BASED ON RHEOLOGICAL AND MICROSTRUCTURAL EVALUATION

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Abstract. Hot-applied joint sealants are widely used to seal cracks and expansion joints in road and bridge pavements. Recycled Crumb Rubber (CRM) derived from end-of-life tires is frequently incorporated to enhance flexibility and deformation resistance while supporting circular economy strategies through waste valorization. However, conventional specification tests (e.g., EN 14188-1) may not fully reflect the influence of rubber dispersion and microstructure on long-term performance. This study evaluates the functional and environmental relevance of CRM in seven commercially available hot-applied joint sealants obtained from five European manufacturers. The materials, including five rubber modified sealants and two comparative sealants, were characterized using fluorescence microscopy and Scanning Electron Microscopy (SEM) to assess phase morphology and rubber distribution. Rheological performance was investigated using dynamic shear rheometry, including Multiple Stress Creep Recovery (MSCR) at 60 °C and Large Amplitude Oscillatory Shear (LAS) at 10 °C. The results demonstrate that sealants compliant with the same specification types (N1/N2) may exhibit markedly different microstructures and viscoelastic responses. Variations in crumb rubber content and filler fraction significantly affected resistance to permanent deformation at high temperature and fatigue performance at low temperature. Materials with a more homogeneous rubber dispersion generally showed improved rheological performance. From an environmental perspective, the effective use of CRM not only diverts waste tires from landfill, but may also improve sealant durability, potentially reducing maintenance frequency and material consumption over the service life of infrastructure systems.

Keywords: hot-applied joint sealant, recycled crumb rubber, rheological and microstructural properties.

1. Introduction

Hot-applied joint sealants are widely used in transportation infrastructure to seal cracks and joints on asphalt and concrete pavements. Their primary function is to ensure watertightness while accommodating cyclic movements induced by traffic load and thermal fluctuations. Sealants used in bridge expansion joints are additionally exposed to complex stress states and repeated dynamic loading, requiring adequate rheological stability and adhesion performance (Di Mascio et al., 2017; Liu et al., 2016). Field investigations have demonstrated that the durability of joints and cracks is strongly correlated with the mechanical properties of the sealant and its resistance to ageing and adhesion loss (Lima & Brito, 2009; Wang et al., 2023).

The functional performance of hot-applied joint sealants, including resistance to permanent deformation at elevated temperatures, flexibility at low temperatures, and retention of adhesion under varying environmental conditions, is directly governed by the composition of the asphalt binder and its modification (Airey, 2004; Cao et al., 2019; Masson et al., 2005). Elastomeric modification is commonly employed to improve rheological performance, particularly through the incorporation of a Crumb Rubber Modifier (CRM) derived from end-of-life tires, often in combination with Styrene-Butadiene-Styrene (SBS). The addition of rubber and polymer modifiers has been shown to improve high-temperature rutting resistance while enhancing elasticity and low-temperature cracking resistance of joint sealants (Gong et al., 2022; Putman & Amirkhanian, 2010).

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The effectiveness of CRM modification depends on the physicochemical interaction between rubber particles and the asphalt phase, including swelling of the rubber, absorption of light fractions, and the resulting changes in microstructure (Putman & Amirkhanian, 2010; Shen & Amirkhanian, 2005). In hot-applied sealants, these interactions are further influenced by thermo-oxidative aging during application and service (Cao et al., 2022).

From an environmental perspective, the incorporation of CRM supports the principles of the principles of circular economy by diverting waste tires from landfills and reducing the demand for virgin raw materials. Life cycle assessment studies indicate that crumb rubber modification may reduce environmental burdens, particularly when implemented via the wet process (Farina et al., 2017; Piao et al., 2022).

In engineering practice, joint sealants are commonly evaluated using empirical specification tests (ASTM D6690 (ASTM International, 2001 and EN 14188-1 (European Committee for Standardization [ECS], 2004), such as cone penetration, softening point, and elastic recovery. However, materials exhibiting similar specification results may differ significantly in phase morphology and viscoelastic behavior (Masson et al., 2005; Zhang et al., 2020). Therefore, advanced rheological techniques, such as Dynamic Shear Rheometry (DSR), Multiple Stress Creep Recovery (MSCR), and Large Amplitude Oscillatory Shear (LAS), combined with microstructural methods including fluorescence microscopy, are increasingly used to assess modifier dispersion and phase organization (Błażejowski et al., 2020; Maciejewski et al., 2025; Shen & Amirkhanian, 2005; Li et al., 2022). These approaches enable differentiation of materials with similar empirical properties but distinct internal structure and damage evolution mechanisms.

In a previous study (Stępień & Remišová, 2023), selected commercial hot-applied joint sealants were characterized using chemical analyzes and empirical tests, revealing substantial differences in composition and rubber fraction morphology. The present study extends that work by examining the relationship between microstructural characteristics and the rheological response in CRM-containing joint sealants.

Unlike previous studies that have focused mainly on laboratory-prepared crumb rubber modified binders, the present work provides a systematic analysis of commercially available hot-applied joint sealants classified as N1 and N2 according to EN 14188-1 (ECS, 2004), providing insight into how actual compositional variability translates into differences in microstructure and rheological response.

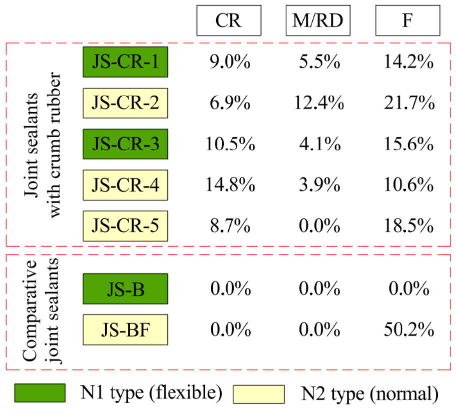
The objective of this study is to determine to what extent the presence and dispersion of recycled crumb rubber influence the viscoelastic response and fatigue performance of hot-applied joint sealants.

2. Materials and methods

2.1. Characterization of analyzed joint-sealants

Seven commercially available hot-applied joint sealants were investigated, compliant with the requirements of EN 14188-1 (ECS, 2004), intended to seal joints and expansion gaps in road and bridge pavements, and sourced from five randomly selected European manufacturers. The materials analyzed included five joint sealants modified with recycled rubber (JS-CR-1 to JS-CR-5) and two comparative sealants without rubber addition (JS-B and JS-BF).

Figure 1 presents the basic material composition of the investigated joint sealants, determined based on extraction procedures.



		CR	M/RD	F
Joint sealants with crumb rubber	🟩 JS-CR-1	9.0%	5.5%	14.2%
	🟨 JS-CR-2	6.9%	12.4%	21.7%
	🟩 JS-CR-3	10.5%	4.1%	15.6%
	🟨 JS-CR-4	14.8%	3.9%	10.6%
	🟨 JS-CR-5	8.7%	0.0%	18.5%
Comparative joint sealants	🟩 JS-B	0.0%	0.0%	0.0%
	🟨 JS-BF	0.0%	0.0%	50.2%

🟩 N1 type (flexible) 🟨 N2 type (normal)

Figure 1. Summary of the material composition of the joint sealants used in the study

The extracted fractions included mineral filler (F), rubber powder/crumb particles larger than 0.19 mm (CR), and a mixed rubber/mineral dust fraction with particle sizes greater than 0.063 mm and less than or equal to 0.19 mm (M/RD).

The presence of recycled rubber particles with varying particle size distributions, origins, and manufacturing processes was identified. Representative SEM images of crumb rubber particles extracted from the analyzed joint sealants, recorded at the same magnification of 200× and including a scale bar of 500 µm, are presented in Figure 2.

Figures 2a and 2c show rubber particles extracted from the N1-type sealants JS-CR-1 and JS-CR-3, respectively, while Figures 2b, 2d, and 2e show rubber particles extracted from the N2-type sealants JS-CR-2, JS-CR-4, and JS-CR-5, respectively. Visual differences in particle size and morphology can be observed among the analyzed samples. Visual inspection indicates that the largest rubber particles were present in Figures 2b and 2e.

The joint sealants investigated (JS-CR-1 to JS-CR-5) contained recycled rubber particles produced using different methods (cryogenic and mechanical) and characterized by varying particle sizes and contents (8.7–19.3%).

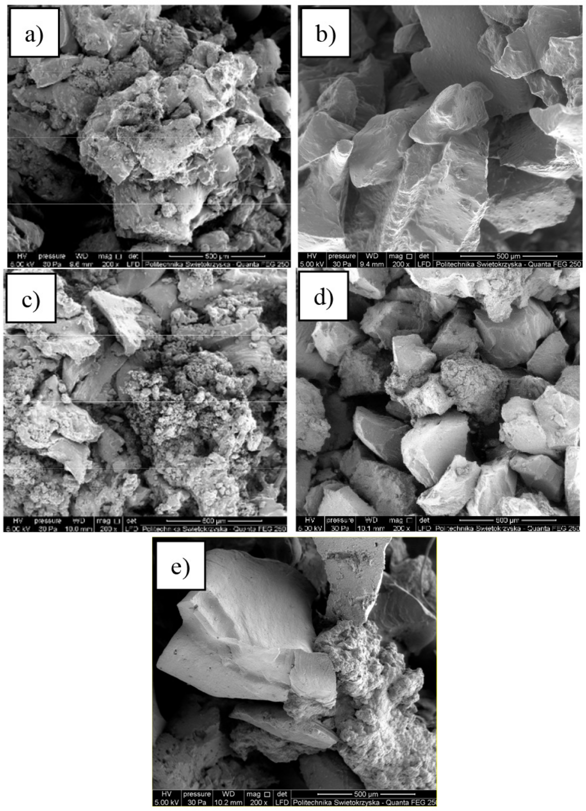


Figure 2. Scanning electron microscope images of rubber powder/crumb used in joint sealant: a) JS-CR-1; b) JS-CR-2; c) JS-CR-3; d) JS-CR-4; e) JS-CR-5

Variations in rubber content and production method are known to influence the physicochemical properties of rubber-asphalt binders (Putman & Amirkhanian, 2010), including viscosity, resistance to high-temperature deformation, and low-temperature performance. Consequently, recycled rubber can be considered a key modifier governing the functional properties of hot-applied joint sealants.

Mineral filler passing through a 0.063 mm sieve was identified in the joint sealants from different manufacturers (JS-CR-1 to JS-CR-5, JS-BF), with contents ranging from 10.6 to 50.2%. Chemical composition analysis performed using EDX spectroscopy indicated that calcium or silicon were the dominant elements, occurring at the highest relative proportions in the analyzed materials.

From a qualitative perspective, the material composition of the joint sealants investigated may influence their potential carbon footprint. The asphalt binder is generally the most emission-intensive component of asphalt-based materials due to the energy-demanding nature of its production process. Mineral filler is characterized by a comparatively lower environmental burden, whereas recycled crumb rubber constitutes a secondary material whose environmental impact is primarily associated with collection and grinding processes. Therefore, sealants with a lower asphalt binder content and a higher proportion of recycled components may potentially exhibit a more favorable environmental performance at the production stage.

The previously reported FTIR results (Stępień & Remišová, 2023) confirmed the multiphase nature of the investigated materials and the presence of elastomeric components in the soluble fractions. FTIR spectra of asphalt binders extracted from joint sealants revealed characteristic absorption bands attributed to butadiene structures, vinyl groups, and styrene structures, confirming the contribution of elastomeric components to the investigated binders.

The joint sealants analyzed exhibited similar elastic recovery values at 25°C according to EN 13398 (91–98%) (ECS, 2010), despite differences in the content and chemical composition of individual components, with the exception of JS-CR-2, for which specimen failure was observed during testing. These results indicate a high degree of elastomeric modification of asphalt binders and/or a pronounced effect of rubber modification, regardless of the type of sealant (N1/N2).

For the binders recovered from JS-CR-2 and JS-CR-5, additional absorption bands attributed to ester functional groups were identified, suggesting the presence of bio-based components. More polar oil-ester constituents may alter the interactions between the binder and the rubber phase (Guo et al., 2024), potentially influencing the degree of rubber swelling and the dispersion of rubber particles within the binder matrix.

2.2. Methods of analysis

Microstructural observations were carried out using reflected-light fluorescence microscopy with a Zeiss optical microscope (Carl Zeiss Microscopy, Germany) equipped with a Zeiss Axiocam MRc 5 camera. The images were acquired at a magnification of 10× under constant illumination conditions. The obtained were used for a qualitative assessment of the microstructural homogeneity and the fluorescence intensity in the investigated joint sealants.

The rheological behavior of the analyzed joint sealants was investigated using a Dynamic Shear Rheometer (DSR) under oscillatory and creep-recovery loading modes. The test program included the following measurements:

Large Amplitude Oscillatory Shear (LAS) tests: Performed at 10 °C using an 8 mm parallel plate geometry to assess material response under increasing strain up to 30%. LAS tests were conducted in accordance with AASHTO TP 101-12. Data were processed using the LAS Analysis Template (Version 1.52) developed by the Modified Asphalt Research Center (MARC), University of Wisconsin-Madison.

Multiple Stress Creep Recovery (MSCR) tests: carried out at 60 °C using a 25 mm parallel plate geometry with successive creep–recovery cycles at shear stress levels of 0.1 and 3.2 kPa.

3. Results

3.1. Fluorescence microscopy analysis

Fluorescence microscopy was used to qualitatively assess the microstructure of the investigated hot-applied joint sealants and to identify differences related to the presence of recycled crumb rubber, the modification type (N1/N2), and the overall composition.

Figure 3 presents fluorescence microscopy images of analyzed joint sealants.

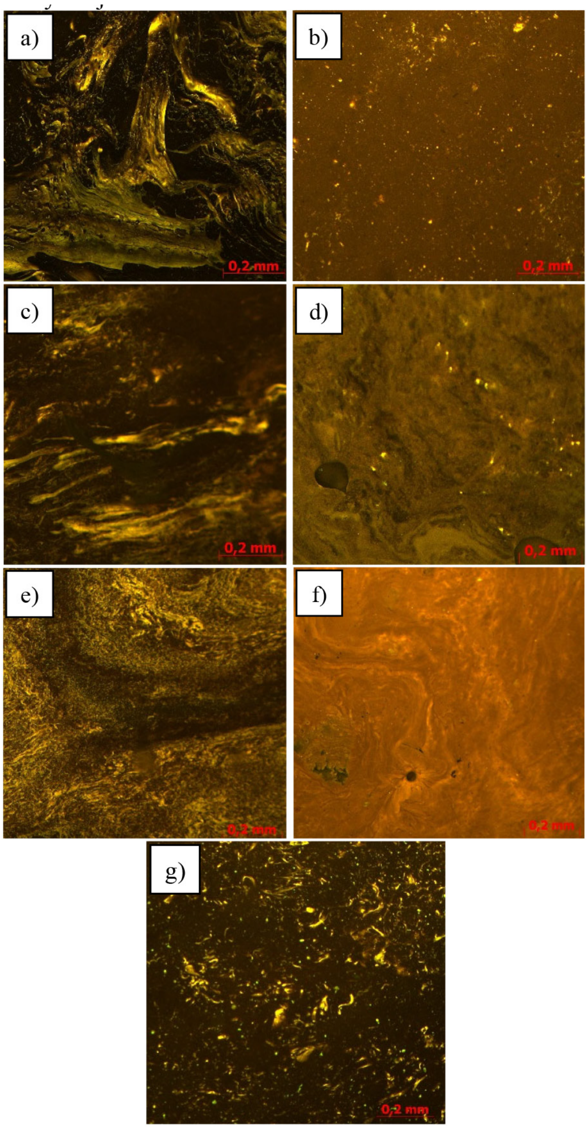


Figure 3. Fluorescence microscopy images of hot-applied joint sealants: a) JS-B; b) JS-BF; c) JS-CR-1; d) JS-CR-2; e) JS-CR-3; f) JS-CR-4; g) JS-CR-5

JS-CR-1 and JS-CR-3 (Figures 3c and 3e) exhibit relatively low phase contrast, with a continuous binder matrix and diffuse regions of varying fluorescence intensity. No well-defined low-fluorescence domains that could be clearly associated with a discrete rubber phase are observed. This may suggest that the rubber fraction is present in a highly swollen and/or finely dispersed form, resulting in poorly resolved phase boundaries. Consequently, the rubber does not appear as a distinct separated secondary phase but rather as a component integrated within the binder matrix, which is consistent with characteristics typically reported for N1-type materials.

In contrast, JS-CR-2 and JS-CR-4 (Figures 3d and 3f) show distinct low-fluorescence domains of varying size and shape dispersed within a fluorescent matrix. These regions can be associated with the rubber fraction acting as a recognizable secondary phase. Locally discernible boundaries suggest limited swelling and/or the presence of larger rubber particles. The microstructure of these materials is more consistent with a classical two-phase system typical of N2-type sealants.

JS-CR-5 (Figure 3g), also classified as N2, presents a predominantly uniform fluorescent background without clearly developed band-like features. Fine, point-like regions of increased fluorescence are visible; however, their morphology does not allow for a definitive assignment to a specific phase. No distinctly separated low-fluorescence domains are apparent at the scale of observation, which may indicate limited visible phase differentiation. This behavior may be related to differences in binder composition compared to the other N2-type materials.

The analysis demonstrates that sealants with similar empirical properties and functional classification may exhibit markedly different microstructures. The differences concern both the dispersion of the rubber and its degree of integration with the binder, indicating that the N1/N2 classification does not necessarily reflect the microstructural organization.

The comparative materials show distinct features. JS-B (Figure 3a), classified as N1, presents a heterogeneous, banded microstructure with flow-like patterns and extensive high-fluorescence regions, suggesting a high content of polymeric or resin-based modifiers. No discrete low-fluorescence domains associated with recycled rubber are observed.

JS-BF (Figure 3b) exhibits a relatively homogeneous, fine-grained binder matrix with localized bright microdomains, which may be related to organic additives.

Fluorescence microscopy analysis demonstrated that joint sealants exhibiting similar empirical test results and comparable functional classifications may nevertheless be characterized by substantially different microstructures. These differences concern both the degree of dispersion of the rubber fraction, as identified by extraction-based methods, and the nature of its integration with the binder. The results confirm that the functional classification of joint sealants (N1/N2) does not always directly reflect the underlying microstructure of the material, and that the spatial distribution and degree of phase integration at the microscopic scale may play an important role in the interpretation of rheological behavior and potential in-service durability.

3.2. Rheological characterization

Rheological tests were conducted to quantitatively assess the viscoelastic behavior of the investigated joint sealants under conditions corresponding to in-service temperature ranges. The use of dynamic rheometry enables detailed characterization of the materials' viscoelastic properties and facilitates the identification of differences arising from compositional and microstructural variations that may not be discernible by conventional empirical tests.

The results of the MSCR test conducted at 60°C are presented in Figures 4–5. Figure 4 shows the values of the non-recoverable creep compliance determined at stress levels of 0.1 kPa and 3.2 kPa. Distinct differences are observed among the sealants, particularly at the higher stress level.

The comparative sealant JS-BF, characterized by a high mineral filler content (50.2%) and the absence of a rubber fraction, exhibits the highest non-recoverable creep compliance values at both 0.1 kPa and 3.2 kPa, with the differences being especially pronounced at 3.2 kPa. This indicates increased susceptibility to creep stressess at higher temperatures. In fluorescence microscopy, this material displayed a relatively homogeneous microstructure without clearly distinguishable regions of contrasting intensity, which may be associated with its distinct rheological response under high-temperature loading conditions.

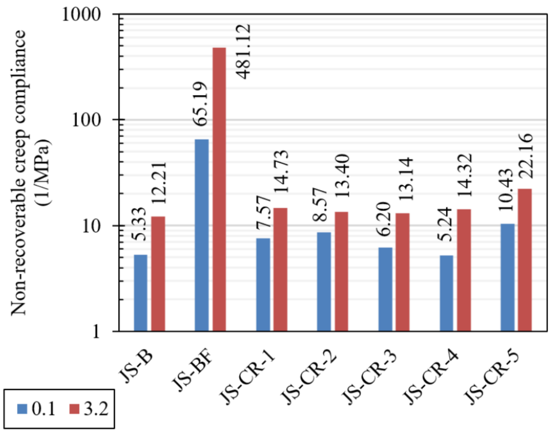


Figure 4. Values of the non-recoverable creep compliance determined in the MSCR test at 60°C at stress levels of 0.1 kPa and 3.2 kPa for the investigated joint sealants

Rubber-modified sealants, particularly JS-CR-3 and JS-CR-4, exhibit distinctly lower values of non-recoverable creep compliance at both stress levels (0.1 kPa and 3.2 kPa) compared to the comparative sealant JS-BF. At the higher stress level (3.2 kPa), these differences become especially pronounced, indicating possibly a greater compatibility of the components of the sealant.

Against this background, the behavior of JS-B should be noted. This sealant does not contain a rubber fraction or mineral filler and is likely based on a highly polymer-modified asphalt binder. At 0.1 kPa, JS-B exhibits non-recoverable creep compliance values comparable to those of JS-CR-4, whereas at 3.2 kPa it reaches the lowest value among all investigated materials. This indicates the highest resistance to permanent deformation at elevated temperatures under higher stress conditions.

In the case of JS-B, the absence of a clearly distinguishable secondary phase in fluorescence microscopy is consistent with a more homogeneous microstructure, typical of materials based on highly polymer-modified binders.

The joint sealants, except JS-B, in this scope represented similar performance, which was caracterized by significantly increased creep compliance (lower stiffness).

Figure 5 presents the percent recovery values determined in the MSCR test. Most of the analyzed sealants, including the rubber-modified materials, achieve high elastic recovery at 0.1 kPa and maintain a relatively high level also at 3.2 kPa. The most pronounced decrease in percent recovery at the higher stress level is observed for JS-BF, which is consistent with its elevated non-recoverable creep compliance values. This indicates a limited ability of this material to recover elastically under increased loading and may suggest reduced compatibility among its constituent components. In contrast, materials exhibiting a more pronounced elastic response maintain high percent recovery at 3.2 kPa, indicating a stable elastic behavior at elevated temperature.

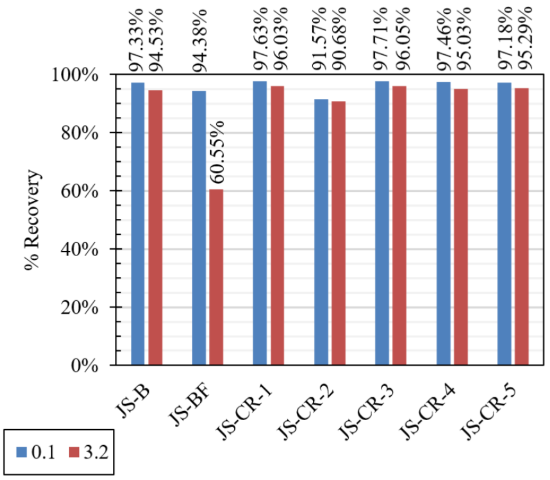


Figure 5. Percent recovery determined in the MSCR test at 60°C for stress levels of 0.1 kPa and 3.2 kPa

The results of the LAS test conducted at 10 °C are presented in Figures 6–7, while the fatigue life indicator values are summarized in Table 1. Figure 6 illustrates the relationship between oscillation stress and strain amplitudes. The highest stress levels within the analyzed deformation range are observed for JS-CR-4 and JS-B, indicating a greater stiffness of these materials under low-temperature and high-strain conditions.

At the same time, sealants classified as N1 type (JS-CR-1, JS-CR-3, and JS-B) exhibit a clearly different curve shape compared to N2-type materials. These characteristics are distinguished by a more gradual increase in the oscillation stress with increasing strain amplitude and a less abrupt transition into the nonlinear range, which may indicate a different mechanism of deformation energy dissipation.

In the case of JS-CR-4, fluorescence microscopy revealed localized regions of reduced fluorescence intensity that may correspond to rubber fraction particles; however, their distribution does not form a clearly separated continuous phase. In contrast, N1-type sealants, in which the fluorescence image exhibited a more homogeneous or banded appearance (e.g., JS-B), demonstrate rheological behavior suggesting a high degree of binder modification and strong integration of organic constituents.

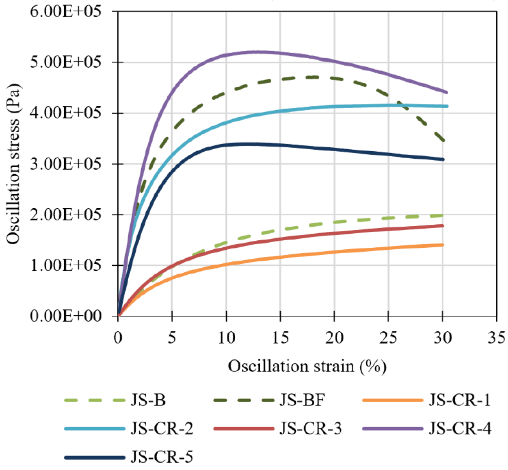


Figure 6. Oscillation stress as a function of oscillation strain determined in the LAS test at 10 °C for the analyzed joint sealants

Figure 7 presents the relationship between the fatigue life indicator (N_f) and strain amplitude. The JS-B sealant exhibits the highest N_f values throughout the entire strain range, confirming its superior resistance to fatigue damage initiation and propagation at low temperature. The curves corresponding to JS-CR-1, JS-CR-3, and JS-CR-4 largely overlap over a wide range of strain amplitudes, indicating comparable fatigue resistance of these materials under low-temperature conditions. On the contrary, JS-CR-2 shows a distinctly different response, characterized by noticeably lower N_f values, particularly at higher strain amplitudes. The joint sealant JS-BF exhibits lower fatigue durability compared to most of the rubber-modified sealants.

The analysis of Figure 7 shows, that the typically evaluated in asphalt binder LAS tests strain levels of 2.5% and 5.0% are not relevant for joint sealants. This Relates to the fact that asphalt binders exhibit far greater strains than asphalt mixtures, with joint sealants being typically a mixture of asphalt binders, fine aggregate particles and other particulate matter fall inbetween these extremes. Hence, a 0.1 to 3% strain levels are evaluated and fatigue life indicators 0.5% and 1.0% strains are calculated in Table 1.

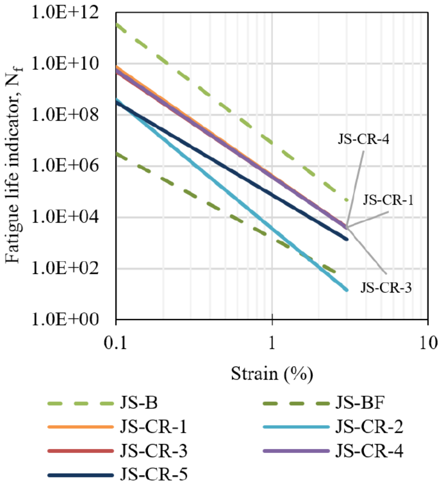


Figure 7. Fatigue life indicator as a function of strain amplitude determined in the LAS test at 10 °C for the analyzed joint sealants

Table 1. Fatigue life indicator determined at 0.5% and 1% strain amplitude and fatigue cracking indicator at 10 °C for the analyzed joint sealants

10 °C	N_f at 0.5% strain	N_f at 1% strain	|G*|·sin(δ) (kPa)
Joint sealant	Fatigue life indicator		Fatigue cracking indicator
JS-B	1,93E+08	7,67E+06	736
JS-BF	1,52E+04	1,55E+03	4722
JS-CR-1	7,78E+06	4,12E+05	674
JS-CR-2	1,20E+05	3,70E+03	4868
JS-CR-3	6,55E+06	3,81E+05	881
JS-CR-4	6,90E+06	3,85E+05	4490
JS-CR-5	9,14E+05	7,37E+04	2817

The data summarized in Table 1 indicate that JS-B achieves the highest N_f values at both 0.5% and 1.0% strain amplitudes, reflecting the highest fatigue resistance under the analyzed conditions. Among the rubber-modified sealants, JS-CR-3 and JS-CR-4 demonstrate relatively favorable performance. The reduced fatigue life indicator observed at higher strain amplitudes (e.g., for JS-CR-2) may suggest lower tolerance to cyclic deformation at low temperature, which may be attributed to differences in the material composition and the spatial distribution of organic constituents within the microstructure.

Table 1 also presents the values of the fatigue cracking parameter $|G^*|\cdot\sin\delta$ determined at 10 °C. According to the criteria of rheological specification, the lower values of $|G^*|\cdot\sin(\delta)$ (e.g., for JS-CR-1 and JS-CR-3) indicate of improved resistance to fatigue crack initiation under repeated loading. On the contrary, higher |G*|·sinδ values (e.g., for JS-CR-2 and JS-BF) reflect a greater complex shear modulus and a lower viscous response, which may correspond to a reduced strain energy dissipation capacity and an increased susceptibility to fatigue cracking.

The combined analysis of the MSCR results (non-recoverable creep compliance, percent recovery) and LAS (oscillatory stress, fatigue life, N_f, and |G*|·sinδ) results confirms that the differences in the composition of the material, particularly the content of crumb rubber and mineral filler fraction – translate into different rheological responses under both elevated temperature conditions and low-temperature cyclic loading.

The evaluated rheological characteristics do not correlate particularly well in the tested scope. Although JS-BF sealant was characterized by poor recovery performance, the worst fatigue characteristics in the LAS test and highest stiffness measured by the |G*|·sinδ parameter, such incidence was not observed in case of the remaining sealants.

Fluorescence microscopy observations reveal significant differences in the spatial organization of organic constituents; however, they do not allow for a direct quantitative correlation between specific microstructural features and individual rheological parameters.

4. Conclusions

Based on the microstructural and rheological investigations conducted on hot-applied joint sealants, the following conclusions can be drawn.

Fluorescence microscopy revealed clear variations in the spatial organization of organic components. Sealants classified as N1 (JS-CR-1, JS-CR-3, JS-B) generally exhibited a more homogeneous or diffuse microstructure, while selected N2 materials (JS-CR-2, JS-CR-4) showed more distinct phase contrast, indicating differences in the dispersion state of crumb rubber and organic components.

The results of the MSCR test at 60 °C confirmed substantial variability in the resistance to high-temperature deformation. The highest non-recoverable creep compliance values were observed for JS-BF (high filler content, no rubber), while JS-B and selected CRM-modified sealants (particularly JS-CR-3 and JS-CR-4) exhibited enhanced resistance to permanent deformation and high percent recovery under both stress levels (0.1 and 3.2 kPa).

LAS testing at 10 °C demonstrated that fatigue performance differed markedly among materials. JS-B exhibited the highest fatigue life indicator values, while JS-CR-1, JS-CR-3, and JS-CR-4 showed comparable fatigue resistance. JS-CR-2 and JS-BF were characterized by lower fatigue performance at higher strain amplitudes.

The combined analysis of the MSCR and LAS parameters indicates that both the crumb rubber content and the level of binder modification significantly influence viscoelastic behavior at elevated and low temperatures. Materials with comparable specification classification (N1/N2) may differ substantially in rheological performance. Since rheological performance is closely related to durability under service conditions, effective utilization of recycled crumb rubber can affect not only functional performance, but also the potential environmental footprint of joint sealants through durability-related effects.

Microstructural observations did not allow for direct quantitative assignment of specific phases to particular rheological parameters; however, they confirmed considerable variability in the dispersion and integration of organic components, which may contribute to differences in mechanical response.

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