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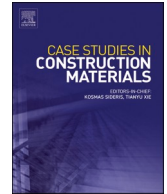
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Effect of Hybrid Surface Treatment of Tire Rubber Aggregates on the microstructure and mechanical properties of Standardized Mortars

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ABSTRACT

The incorporation of tire rubber aggregates in cement-based mortars reduces the consumption of natural sand but typically leads to substantial strength losses because of the weak and porous interfacial transition zone (ITZ) formed around untreated rubber. This study develops a hybrid mineral-epoxy surface treatment designed to overcome this limitation by forming a continuous composite shell composed of sand, cement, silica fume and epoxy resin. Microstructural analyses show that the treatment yields a uniformly coated rubber surface, a denser particle morphology and a markedly refined ITZ: the interfacial layer around untreated rubber is wide and highly porous ($\approx 14\text{--}15\ \mu\text{m}$), whereas the hybrid coating reduces it to a compact band of about $1\text{--}2\ \mu\text{m}$. These refinements translate into consistent mechanical gains across all curing ages, with flexural strength increasing by 11–27% and compressive strength by 23–76%, depending on substitution level and age. In parallel with these mechanical and microstructural improvements, the method offers a favourable cost-performance balance, particularly thanks to the possibility of reducing resin consumption through optimisation of the adhesive film. The hybrid coating concept significantly strengthens the feasibility of producing high-performance and economically viable rubberized mortars and, ultimately, rubberized concretes.

1. Introduction

The accumulation of waste tires has become a global environmental and economic issue, with more than 1.5 billion units discarded annually [1]. Improper management leads to landfilling and environmental hazards [2]. In the context of a circular economy, recovering and reusing end-of-life tires is therefore a pressing necessity [3]. The construction sector, one of the largest consumers of natural resources, offers a promising outlet for such reuse [4,5]. Incorporating crumb rubber into cementitious materials can enhance impact resistance, energy absorption, and vibration damping [6–8]. However, its broader structural application remains limited due to the low stiffness and hydrophobic surface of rubber, which lead to poor bonding with cement and the formation of a weak interfacial transition zone (ITZ) [9–15]. This interface encourages premature stress concentration and cracking, compromising the material's strength relative to traditional mixes [16,17]. Addressing these adhesion issues is vital for creating sound rubber-modified

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cementitious materials.

Various techniques have been explored to improve rubber-cement bonding, such as chemical etching, acid or base treatments, coating applications, and exposure to radiation or specific agents [18–26]. While these methods can enhance adhesion, their reliability varies, and some involve multi-step processes or hazardous substances, limiting their scalability and sustainability [27–31]. In addition, many previous studies have evaluated mechanical performance without systematically investigating how surface treatments modify the ITZ, resulting in an incomplete understanding of the mechanisms responsible for strength loss. Moreover, many studies have focused on single-action treatments and have not adequately addressed the persistent weakness of the rubber-cement interface, which remains the central scientific challenge. These limitations indicate that current approaches fail to provide a robust and scalable solution to the adhesion problem, leaving a clear research gap regarding a scalable treatment that can simultaneously address both chemical compatibility and mechanical anchorage.

Recent work has been done on multi-component coatings to address both adhesion and mechanical interlocking [32–34]. Cementitious or mineral coatings may improve cement hydration and reduce interfacial voids; these systems often require additional processing, which complicates replication and manufacturing. Furthermore, limited research has provided direct correlations between microstructural refinement at the interface and corresponding mechanical improvements [35], leaving uncertainties about how such coatings influence ITZ performance. This gap highlights the need for a simple treatment capable of simultaneously improving chemical compatibility and mechanical anchorage, while remaining compatible with standard mixing procedures. Therefore, the scientific problem that remains unresolved is the development of a practical and reproducible hybrid treatment that can correct the fundamental incompatibilities between hydrophobic rubber and hydrophilic cementitious matrices.

Previous studies have consistently shown that replacing natural aggregates with waste tire rubber significantly reduces strength because of the hydrophobic and deformable nature of rubber, which limits adhesion with the cement paste and weakens the interfacial zone [36–40]. Similar observations across rubberised mortars and concretes confirm the inherent vulnerability of the interfacial zone. Traditional surface modifications, such as NaOH washing [41], UV modification [42,43], silane coupling [44], epoxy coatings [45], or mineral coatings [46–48], have generally produced modest improvements, rarely exceeding 15 % in compression and 10 % in flexure. These limited gains reflect the fact that most treatments activate only one interfacial mechanism and therefore fail to adequately address the combined chemical and mechanical incompatibilities. Thus, despite the extensive literature, the fundamental issue of insufficient interfacial bonding remains unresolved, confirming the scientific relevance of developing a more comprehensive surface treatment strategy. In particular, no previous work has demonstrated a coating approach capable of integrating adhesion, mechanical interlocking, and pozzolanic refinement in a single and operationally simple process.

In this study, a hybrid surface treatment combining epoxy resin, silica fume, cement, and fine sand was developed to form a multifunctional coating that enhances both chemical compatibility and mechanical anchorage. In this formulation, the epoxy resin ensures the adhesion of the mineral fines (cement, silica fume, and fine sand) onto the rubber surface, while the mineral particles increase surface roughness and promote additional hydration. Together, these components act synergistically to create a denser and more cohesive interfacial transition zone. The novelty of this approach lies in the use of a one-step Hybrid surface treatment that simultaneously improves wettability, mechanical interlocking, and microstructural densification, without requiring additional processing steps or hazardous treatments.

This paper examines a single-step surface treatment with epoxy resin, cement, silica fume, and fine sand. The intention is to enhance surface roughness, promote cement hydration, and strengthen rubber-cement adhesion. Designed for simplicity and compatibility with normal mixing procedures, this treatment avoids the complexity of previous methods. To evaluate its effectiveness, standard cement mixes were prepared with 25 %, 50 %, and 75 % sand replacement by rubber, with and without the treatment. Flexural and compressive strength tests were conducted after different curing periods. Microscopy was employed to analyse the interfacial microstructure. In addition, this work introduces two aspects often overlooked in earlier studies: a cost-performance evaluation to assess the economic relevance of the treatment, and a dedicated section on limitations and recommendations to clarify the scope of the results and identify necessary future investigations. The explicit linkage between microstructural evidence, interfacial chemistry and mechanical performance clarifies how the hybrid surface treatment improves ITZ behaviour, and addresses the scientific gap identified above regarding the relationship between surface modification, ITZ enhancement and global mechanical performance. These findings support the development of more reliable and sustainable rubber-modified cementitious materials and suggest a practical way of integrating recycled rubber into cementitious composites with improved performance. The treatment also offers a technically viable means of using waste tyres in construction applications [32–34].

2. Materials and methods

This section presents the materials used in the study, the hybrid surface treatment developed for the rubber aggregates, and the procedures applied for preparing the standardized mortars. The methodology is organized to clearly distinguish the characterization of raw materials, the coating process applied to the rubber particles, and the fabrication of the different mortar mixtures. All experimental steps were conducted following controlled and reproducible procedures to ensure consistency across batches and reliability in the subsequent mechanical and microstructural analyses.

2.1. Materials and procedure used for the hybrid surface treatment

The hybrid surface treatment was applied to commercial SBR rubber aggregates supplied by Delta-Gom (France) [49]. These aggregates consist of mechanically shredded end-of-life tyres collected through the Aliapur recycling chain, with a nominal particle

size of 1–4 mm and a bulk density of approximately 500 kg/m^3 . According to the manufacturer's technical data sheet, they contain no textile or metallic contaminants and exhibit the typical irregular and angular morphology of mechanically processed tyre rubber. This surface texture, combined with the hydrophobic nature of SBR, is known to reduce adhesion with cementitious matrices and therefore motivates the use of the hybrid coating system investigated in this study.

The hybrid surface treatment applied to the rubber aggregates required an additional set of materials distinct from those used in the mortar mixtures. This treatment involved a low-viscosity epoxy resin serving as an adhesive phase, a fine sand fraction providing mechanical asperities, Portland cement acting as a reactive hydraulic binder, and silica fume supplying ultrafine pozzolanic particles, as illustrated in Fig. 1, which shows the materials and equipment used as well as the appearance of the aggregates before and after coating. All materials used in this procedure complied with the relevant standards, including EN 196–1 for sand [50] and EN 197–1 for cement, and were selected to ensure full compatibility with the mortar formulation.

The Hybrid surface treatment was formulated using four components: S&P Resin 55 HP, fine EN 196–1 sand (0–0.5 mm), CEM I 52.5 N cement and silica fume Condensil S-95 DP [51]. Their physical and mechanical characteristics are summarised in Table 1. The viscosity of the resin allowed it to wet the rubber surface effectively before the mineral phase was applied, ensuring continuous coverage. The mineral constituents were selected to provide full compatibility with the cementitious matrix and to promote the formation of a dense interfacial layer once the coating hardened.

To optimise the granular packing of the mineral fraction applied onto the rubber surface, a complementary experimental study was performed on mixtures combining fine sand, cement and silica fume. Bulk density, real density and the resulting compacity were measured to identify the most efficient blend. As summarised in Table 2, four combinations were evaluated, and the mixture containing 50 % fine sand, 40 % cement and 10 % silica fume exhibited the highest compacity (0.60). This denser granular skeleton ensures a more uniform mineral distribution around the rubber particles and enhances the mechanical interlocking capacity of the Hybrid surface treatment. Consequently, this optimised blend was adopted for the coating formulation used in the present study. This result is also consistent with published findings on high-performance cementitious materials, where silica fume contents around 10–15 % have been shown to maximise packing density and enhance strength development through improved particle packing and secondary C-S-H formation [52].

A total of 20 kg of untreated rubber aggregates were introduced into a high-shear planetary mixer, whose intense and uniform shear field prevents any risk of particle agglomeration, in one batch, a quantity chosen to ensure efficient torque transfer and homogeneous coating within the equipment's capacity. The treatment followed a continuous mixing sequence designed to modify both surface texture and chemical reactivity. The dry rubber was first homogenised for 2 min, after which a low-viscosity epoxy resin was progressively sprayed, introduced gradually over approximately one minute during rotation, and mixing continued to complete a total of three minutes, forming a thin adhesive film around each particle. The epoxy dosage was predetermined to produce an adhesive coating of approximately $25 \mu\text{m}$, a value consistent with the mean particle diameter of the rubber aggregates, which is about 2 mm, and with their real density of 1 g/cm^3 . These parameters ensured that the applied resin quantity generated a continuous film around each particle.

Once the resin film was formed, a pre-blended mineral mixture of fine sand, cement and silica fume was added, also introduced gradually over roughly one minute, followed by continued mixing to complete a total of three minutes, ensuring that the coating adhered uniformly to the resin layer. Several quantities of the mineral blend (5, 6 and 7 kg per 20 kg of rubber) were assessed within this continuous mixing sequence, and the 6-kg dosage was selected because it provided full and uniform coverage without leaving unbonded residue. Fine sand contributed angular asperities that improved roughness, cement supplied hydration products that strengthened the interface, and silica fume filled micro-voids and enhanced coating density. The use of epoxy resin was motivated by its ability to penetrate micro-irregularities on the rubber surface and to establish a stable mechanical interface once polymerised, despite the hydrophobic nature of rubber.

After mixing, the coated aggregates were spread as a thin and uniform layer over a wide polyethylene sheet to prevent sticking and to ensure maximum exposure to air, inside a controlled curing room maintained at $23 \text{ }^\circ\text{C}$ and 50 % relative humidity. This arrangement ensured that each particle remained fully separated and continuously ventilated throughout the curing period. According to the S&P Resin 55 HP technical data sheet, the system requires a final curing time of seven days to achieve complete polymerisation and full mechanical stabilisation of the epoxy film. The aggregates were therefore kept under these controlled conditions for the entire seven-day curing period, ensuring optimal cross-linking and complete hardening of the resin. After curing, the aggregates were sieved through a 1-mm mesh to remove loose, non-adherent mineral particles, without altering the integrity of the coating, and then stored in sealed containers prior to their incorporation into the mortar mixtures.

Fig. 2 shows the density test performed to assess floatability. The procedure consisted of introducing 300 g of untreated or treated rubber aggregates into a 1-L graduated cylinder containing 700 mL of water. The untreated rubber floated, raising the water level to 1000 mL, which corresponds to a real density of 1.00 g/cm^3 . In contrast, almost all treated particles settled at the bottom, with a final water level of 955 mL. The difference of 255 mL in displaced volume corresponds to a real density of 1.18 g/cm^3 . This simple sedimentation test confirms the increase in density resulting from the mineral coating.

The Hybrid surface treatment system relies on complementary mechanisms. The epoxy resin provides mechanical adhesion and anchorage points for the mineral phase. Cement hydrates after incorporation into the mortar and contributes to a coherent interfacial structure. Silica fume reacts pozzolanically with calcium hydroxide to densify the interface. Fine sand contributes mechanical interlocking through its angular geometry. These combined mechanisms significantly reduce the incompatibility between hydrophobic rubber and the hydrophilic cement matrix.

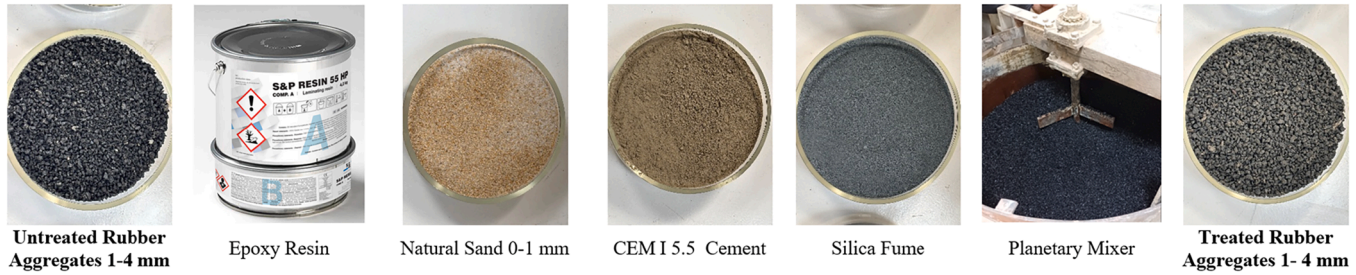


Fig. 1. Materials and equipment used for the hybrid surface treatment of rubber aggregates, and visual appearance of the aggregates before and after coating.

Table 1
Material properties used in the Hybrid surface treatment.

Material	Key properties
Epoxy resin (S&P Resin 55 HP)	Density: 1.0 g/cm ³ ; viscosity: 450–650 mPa·s; tensile strength: 55 MPa; elastic modulus: 2.9 GPa
Fine sand 0–0.5 mm (EN 196–1 standardized)	Nature: standardised siliceous sand; specific gravity: 2.64 g/cm ³
Cement (CEM I 52.5 N)	Density: 3.15 g/cm ³ ; Blaine fineness: 3500 cm ² /g; 28-day compressive strength: 62 MPa
Silica fume (Condensil S-95 DP)	SiO ₂ content: 95 %; BET surface: 22.5 m ² /g; real density: 2.24 g/cm ³ ; bulk density: 0.64 g/cm ³ ; high pozzolanic reactivity

Table 2
Optimisation of the packing properties of fine mineral blends used for the Hybrid surface treatment.

Mix ID	Fine sand (%)	Cement (%)	Silica fume (%)	Bulk density (g/cm ³)	Real density (g/cm ³)	Compacity
Mix A	50	30	20	1,46	2,69	0,54
Mix B	50	40	10	1,68	2,79	0,60
Mix C	50	35	15	1,52	2,74	0,55
Mix D	70	20	10	1,57	2,71	0,58



Fig. 2. Density and wettability of untreated and treated rubber aggregates.

2.2. Materials and preparation of mortar mixtures

The mortars produced in this study were composed of Portland cement, standardized siliceous sand, and crumb rubber aggregates, used either in their untreated form or after hybrid surface treatment. The binder was a CEM I 52.5 N Portland cement (Durabat Xtrem, Lafarge) [44] compliant with EN 197–1. According to its technical specifications, this cement has a real density of 3.15 g/cm³ , a Blaine fineness of approximately 3500 cm²/g, and a 28-day compressive strength of 62 MPa, with sulphate and chloride contents remaining within the limits required for structural applications.

The fine aggregate was a standardized siliceous sand conforming to EN 196–1 and ISO 679. It consisted mainly of clean, rounded quartz grains with a specific gravity of 2.64 g/cm^3 . The sand was supplied in controlled granulometric fractions satisfying the cumulative refusal requirements prescribed by EN 196–1, ensuring highly reproducible particle-size distribution.

The crumb rubber aggregates incorporated into the mortars correspond to the same 1–4 mm SBR fraction described previously. For mixture design purposes, the densities measured for the untreated and hybrid-treated aggregates were 1.00 g/cm^3 and 1.18 g/cm^3 , respectively, and were used to determine the mass of rubber required for each volumetric substitution level. Fig. 3 presents the materials and equipment used for mortar production.

Mortars were prepared in accordance with EN 196–1 by partially replacing natural sand with untreated (MR) or hybrid-treated (MTR) rubber aggregates at volumetric substitution levels of 25 %, 50 %, and 75 %. A reference mortar (NM) was prepared according to EN 196–1, with a fixed sand-to-cement ratio of 3:1 and a constant water-to-cement ratio ($w/c = 0.50$). The substitution was performed on a volumetric basis to maintain equivalent total aggregate volumes, accounting for the lower density of rubber compared with sand. The mass of rubber for each mixture was calculated from its measured density and adjusted to preserve a constant total aggregate volume and binder content. In practice, the reference sand mass per batch was 1350 g. For each replacement level, 25 %, 50 %, or 75 % of this sand mass was replaced by an equivalent volume of rubber granules, determined from the density ratio. Table 3 presents the mix proportions of all mortars, including the reference mix (NM) and the rubberized mixtures containing either untreated (MR) or hybrid-treated (MTR) rubber aggregates.

The substitution of natural sand by rubber caused a progressive decrease in the fresh bulk density as the replacement level increased. For mortars with untreated rubber aggregates, the fresh bulk density dropped from 2301 kg/m^3 for the reference mix to 2037, 1786, and 1552 kg/m^3 for 25 %, 50 %, and 75 % substitution, respectively. This reduction reflects the low specific gravity of rubber aggregates (1.00 g/cm^3) compared with natural sand (2.64 g/cm^3), resulting in lighter mortars and slightly higher air content due to the hydrophobic and poorly wettable surface of untreated rubber.

In contrast, mortars containing hybrid-treated rubber aggregates exhibited slightly higher bulk densities at equivalent replacement levels (2088, 1870, and 1641 kg/m^3 for 25 %, 50 %, and 75 % substitution, respectively). This improvement is attributed to the higher apparent density of the treated rubber (1.18 g/cm^3) and the enhanced adhesion and packing efficiency provided by the mineral-epoxy coating. The surface treatment strengthens the bond with the cement paste, reduces interparticle voids, and produces denser and more cohesive fresh mixes.

Although no direct workability measurements (e.g., flow-table tests) were performed, all mixtures were prepared according to EN 196–1, which ensures consistent and workable mortars through the use of a fixed w/c ratio of 0.50 and standardized mixing and compaction procedures. Workability was therefore indirectly controlled through compliance with the norm and through consolidation on a shock table, and no casting difficulties were observed.

All mortars were prepared and cast in accordance with EN 196–1 (2016). The dry components were first mixed for 2 min in mixer. Water was then added gradually, and mixing continued for 3 min to obtain a homogeneous paste, with rubber aggregates introduced during the final minute to ensure uniform dispersion. The fresh mortar was poured into three-cavity molds ($40 \times 40 \times 160 \text{ mm}$) in two layers, each compacted on a vibrating table for 60 s to remove entrapped air. After casting, the moulds were stored at $20 \pm 2 \text{ }^\circ\text{C}$ and 95 % relative humidity for 24 h. The specimens were then demoulded and cured in lime-saturated water maintained at $20 \pm 2 \text{ }^\circ\text{C}$ until mechanical testing (7, 14, 28, and 90 days).

Fig. 4 presents the particle-size distribution curves of the natural standard sand (NSS), the untreated rubber (UR), the treated rubber (TR), and the sand-rubber blends prepared at 25 %, 50 %, and 75 % volume substitution. The ASTM C33 grading limits and the ideal Fuller curve are also included for reference.

The natural sand displays a continuous, well-graded particle-size distribution that fits entirely within the ASTM C33 envelope. In contrast, both untreated and treated rubber aggregates exhibit a much coarser and highly discontinuous grading, characterised by the absence of fine particles and a steep increase in the coarse fraction. The grading curves of UR and TR remain very similar; the only clear difference is a slight shift toward larger particle sizes for the treated rubber, caused by the mineral coating that increases its apparent grain size.

When rubber is incorporated into the sand-rubber blends, the overall grading progressively changes according to the substitution level. At 25 % and 50 % volume substitution, the blends remain close to or partially within the ASTM C33 limits because the natural sand still governs the distribution. However, at 75 % substitution, the blends clearly fall outside the ASTM envelope, indicating that high rubber contents strongly disrupt grading continuity. Treated and untreated blends exhibit nearly identical distributions, with only a small shift toward coarser sizes for the treated mixtures.

Comparison with the Fuller curve shows that all blends deviate increasingly from the ideal continuous grading as the rubber content increases. This trend reflects the intrinsic lack of fines and the discontinuous nature of rubber gradation, which cannot be compensated by adjusting the substitution level. The 25 % and 50 % blends remain closest to the Fuller curve, suggesting relatively acceptable packing behaviour, whereas the 75 % blends diverge markedly, indicating poor packing density and increased void content. The slight shift associated with the hybrid treatment does not alter this trend, confirming that the substitution level rather than the surface treatment controls the deviation from the ideal grading.

In summary, the results clearly show that the hybrid surface treatment does not modify the fundamental grading profile of rubber aggregates; it only increases their apparent particle size. The dominant parameter governing the packing quality and conformity to ASTM limits is the volume fraction of rubber introduced into the mixture.



Fig. 3. Materials and equipment used for mortar production.

Table 3

Mix proportions of rubberized mortars.

Mix ID	Sand replacement (%)	Type of rubber aggregate	Cement (g)	Sand (g)	Rubber (g)	Water (g)	w/c ratio	Fresh bulk density (kg/m ³)
NM	0	—	450	1350	—	225	0.5	2301
MR25	25	Untreated	450	1012	115	225	0.5	2037
MR50	50	Untreated	450	675	230	225	0.5	1786
MR75	75	Untreated	450	337	345	225	0.5	1552
MTR25	25	Hybrid-treated	450	1012	136	225	0.5	2088
MTR50	50	Hybrid-treated	450	675	272	225	0.5	1870
MTR75	75	Hybrid-treated	450	337	408	225	0.5	1641

2.3. Experimental methods

The experimental program combined microscopic characterization, elemental analysis and mechanical testing to characterise both the microstructural features and the mechanical response of the mortars. The following procedures were applied systematically to all mixtures.

The morphology and surface condition of untreated and hybrid-treated rubber aggregates were examined using a Keyence VHX-7000 digital microscope, with observations focusing on surface texture, coating uniformity and micro-roughness as key indicators of the treatment's effectiveness. The microstructure and mineralogical composition of the hardened mortars were analysed using a JEOL JSM-7100F scanning electron microscope equipped with an Oxford Instruments EDS/EBSD system (AZtec HKL Advanced, Nordlys Nano detector). Energy-dispersive spectroscopy (EDS) was performed to determine the elemental composition across the rubber-matrix interface at an accelerating voltage of 20 kV and an acquisition time of 60 s, with spectra collected in the rubber, matrix, and interfacial transition zone (ITZ) regions to ensure repeatability.

Mechanical testing was carried out at 7, 14, 28 and 90 days to assess the development of mechanical properties. Flexural and compressive strengths were determined in accordance with EN 196-1 [53]. All prismatic specimens (40 × 40 × 160 mm) were demoulded after 24 h and immediately placed in water maintained at 20 ± 2 °C, where they remained fully submerged until the designated testing age. Flexural loading was applied on three prisms, and the six fractured halves obtained from them were subsequently used for the compressive strength determinations. The experimental programme included the reference mortar (NM), the untreated rubber mortar (MR) and the hybrid-treated rubber mortar (MTR). Compressive loading was applied at a constant rate of 2.4 kN·s⁻¹, as prescribed by EN 196-1, until failure.

This combined mechanical and microscopic investigation clarifies how interfacial morphology influences mechanical response and identifies the bonding mechanisms responsible for the enhanced performance of mortars containing hybrid-treated rubber aggregates.

3. Results and discussion

This section presents the experimental results on the hybrid surface treatment of tire rubber aggregates and their incorporation into cement mortars. The discussion follows the logical sequence of the study. First, the morphology and microstructural features of the untreated and treated rubber aggregates are examined to determine how the Hybrid surface treatment modifies surface roughness, texture and reactivity, and to evaluate its potential to enhance adhesion with the cement paste. Second, the microstructure of the hardened mortars is analysed, with particular emphasis on the interfacial transition zone surrounding the rubber particles, in order to assess how the surface modification influences matrix densification and interfacial continuity. Third, coupled SEM-EDS analyses are used to clarify the chemical composition of the cement paste, the treated aggregates, and the ITZ, and to identify the physicochemical mechanisms responsible for improved bonding. Finally, the mechanical performance of the mortars is evaluated through flexural and compressive strength tests, and the observed trends are interpreted in light of the microstructural and chemical evidence. Together, these results clarify how the hybrid surface treatment affects interfacial quality, microstructure and mechanical behaviour in rubberised mortars.

3.1. Surface morphology and microstructural characterization of untreated and treated rubber aggregates

Before incorporation into the mortar, the rubber aggregates were analysed in order to determine how their initial surface state and the hybrid surface treatment influence morphology and adhesion with the cement paste. A three-dimensional optical profilometer was first employed to quantify surface topography by extracting numerical roughness parameters, and microstructural observations were then performed using scanning electron microscopy. Small fragments of fractured mortar, approximately 5 mm in size, were collected after mechanical testing, vacuum-dried and coated to ensure conductivity.

Surface-roughness measurements were conducted in accordance with ISO 4287, which defines the roughness parameters, and ISO 4288, which governs measurement procedures. The profilometer provides the arithmetic mean roughness Ra, representing the average deviation of the profile from the mean line, while the roughness-height parameter Rz expresses the mean height of the major surface irregularities. The parameter RMS corresponds to the quadratic mean roughness Rq, which is more sensitive to extreme peaks and depressions. The maximum peak height Rp and maximum valley depth Rv complete the quantitative description of the surface topography.

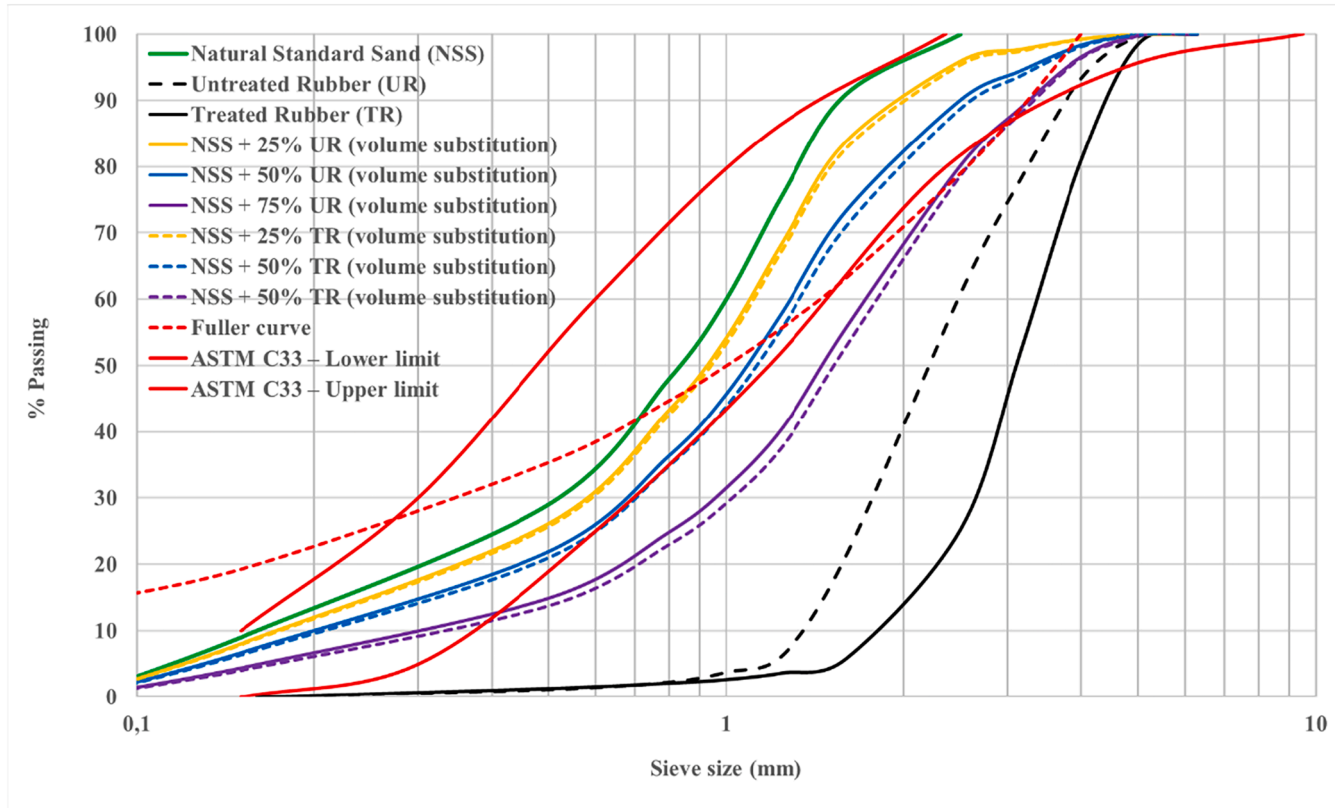


Fig. 4. Particle-size distribution of sand, rubber, and sand-rubber blends compared with ASTM C33 and Fuller.

The untreated rubber shows a very smooth and weakly textured surface. In Fig. 5a, the particle appears dark, flat and almost featureless, a morphology typical of mechanically ground tire rubber. The profilometer (Fig. 5b) confirms this observation, with $R_a = 31.60 \mu\text{m}$, $R_z = 120.58 \mu\text{m}$, $RMS (R_q) = 63.69 \mu\text{m}$, $R_p = 56.90 \mu\text{m}$ and $R_v = 63.69 \mu\text{m}$. These low amplitudes indicate limited asperities and shallow irregularities. Such smoothness restricts mechanical anchorage, reduces cement-paste wetting and weakens the interfacial transition zone once the aggregates are incorporated into the matrix.

The hybrid-treated aggregate presents a radically transformed surface. As shown in Fig. 6a, a continuous and bright epoxy film uniformly covers the rubber. This glossy layer ensures complete coating. Embedded within the resin, fine sand grains form angular yellow protrusions rising sharply from the surface. Very small cement and silica-fume particles densely adhere to the resin and fill the microcavities, giving the surface a granular, mineralised appearance. The profilometer measurements (Fig. 6b) reveal the magnitude of this transformation: $R_a = 550.03 \mu\text{m}$, $R_z = 2059.98 \mu\text{m}$, $RMS (R_q) = 821.56 \mu\text{m}$, $R_p = 821.56 \mu\text{m}$ and $R_v = 1238.42 \mu\text{m}$. Compared with untreated rubber, the surface amplitude increases by more than one order of magnitude. The strong rise in R_a and R_z reflects the formation of deep depressions and large asperities, while the increase in R_q confirms the presence of sharp peaks capable of mechanical interlocking. Sand grains contribute to micro-anchoring, and the fine cementitious particles provide a mineral surface compatible with the cement paste.

Complementary SEM observations of the untreated and hybrid-treated rubber aggregates (Fig. 7 and 8) provide direct morphological evidence supporting the roughness measurements. The untreated rubber surface exhibits the typical characteristics of mechanically shredded tire particles: a relatively smooth polymer skin with elongated ridges, shallow depressions and scattered debris from the cutting process. At $500\times$ magnification, the surface presents continuous micro-ridges separated by gently sloping valleys, confirming the limited asperity development. At $1000\times$ magnification, fine cracks, local tearing of the polymer matrix and occasional voids become visible, yet the overall texture remains mostly flat and non-reactive. Such a surface offers minimal mechanical interlocking and provides no sites for the nucleation or retention of cementitious phases, explaining the weak adhesion generally observed between untreated rubber and cement paste.

The hybrid-treated rubber aggregates display a completely different morphology. At $500\times$ magnification, the surface is uniformly covered by a continuous mineral coating formed during the single-step treatment. This shell consists of epoxy resin acting as a binder and of a dense assemblage of fine sand grains, cement particles and silica-fume clusters mechanically anchored to the polymer substrate. The resulting surface shows highly irregular and multi-scale roughness with sharp asperities, angular protrusions and numerous potential contact points enhancing mechanical anchorage within the cement matrix. At $1000\times$ magnification, the coating appears compact, cohesive and well distributed. Siliceous grains and fine cement particles are intimately embedded within the thin epoxy film, forming a heterogeneous but continuous crust. Because the aggregates have not yet been mixed with water or cement, no hydration products are present at this stage. The coating thus consists entirely of unhydrated mineral particles firmly attached to the resin, providing both chemical compatibility (due to cement and silica fume) and enhanced physical interlocking through the angular morphology of the fine sand. This mineral-rich and roughened surface stands in stark contrast with the smooth polymer baseline observed in untreated aggregates.

Profilometry and SEM observations consistently show that the hybrid surface treatment transforms rubber from a smooth and hydrophobic polymer into a rough, mineral-coated and chemically receptive surface. The treated aggregates exhibit a continuous shell composed of angular sand grains, cement particles and silica-fume clusters firmly anchored to the epoxy film, generating pronounced multi-scale asperities and numerous potential bonding sites. This combined mechanical and chemical enhancement promotes stronger adhesion and facilitates the nucleation of hydration products once the aggregates are embedded in the cement paste. As a result, the Hybrid surface treatment enables the formation of a denser and more cohesive interfacial transition zone, which provides the microstructural basis for the improved mechanical performance observed in mortars incorporating hybrid-treated rubber particles.

3.2. Coupled mechanical and microstructural assessment of rubberized mortars

Following the physical and chemical characterization of the aggregates, this section examines the mechanical behaviour of rubberized mortars and explains the observed trends through a detailed microstructural analysis. The results of flexural and compressive strength tests are first presented to quantify the effect of untreated and hybrid-treated rubber particles on the mechanical performance of the mortar. These macroscopic behaviours are then interpreted through SEM and SEM-EDS observations, with particular emphasis on the morphology and chemical continuity of the interfacial transition zone (ITZ). This combined mechanical-microstructural approach provides a coherent understanding of how the hybrid surface treatment enhances bonding, densifies the ITZ, and ultimately improves the global behaviour of the material.

The mechanical performance of mortars incorporating untreated and hybrid-treated rubber aggregates was evaluated through standardized flexural and compressive strength tests in accordance with EN 196-1, and interpreted in the light of SEM and SEM-EDS observations of the interfacial transition zone (ITZ). The objective was to assess the extent to which the proposed hybrid surface treatment compensates for the mechanical losses typically associated with rubber incorporation and to clarify the microstructural mechanisms governing these trends.

The evolution of flexural strength with curing time is shown in Fig. 9. Mortars containing untreated rubber aggregates exhibited a consistent reduction in strength compared with the reference mortar at all ages. At 7 days, the reference reached 7.5 MPa, whereas MR25, MR50 and MR75 attained 4.5, 3.1 and 2.6 MPa, corresponding to reductions of approximately 40 %, 59 % and 65 %, respectively. At 14 days, the reference mortar reached 7.9 MPa, while MR25, MR50 and MR75 achieved 4.8, 3.2 and 2.6 MPa. At 28 days, the reference reached 8.6 MPa compared with 4.9, 3.2 and 2.6 MPa for the three rubberised mixes. At 90 days, the reference attained 9.2 MPa compared with 5.1, 3.5 and 2.8 MPa for MR25, MR50 and MR75, respectively. These reductions, which intensify

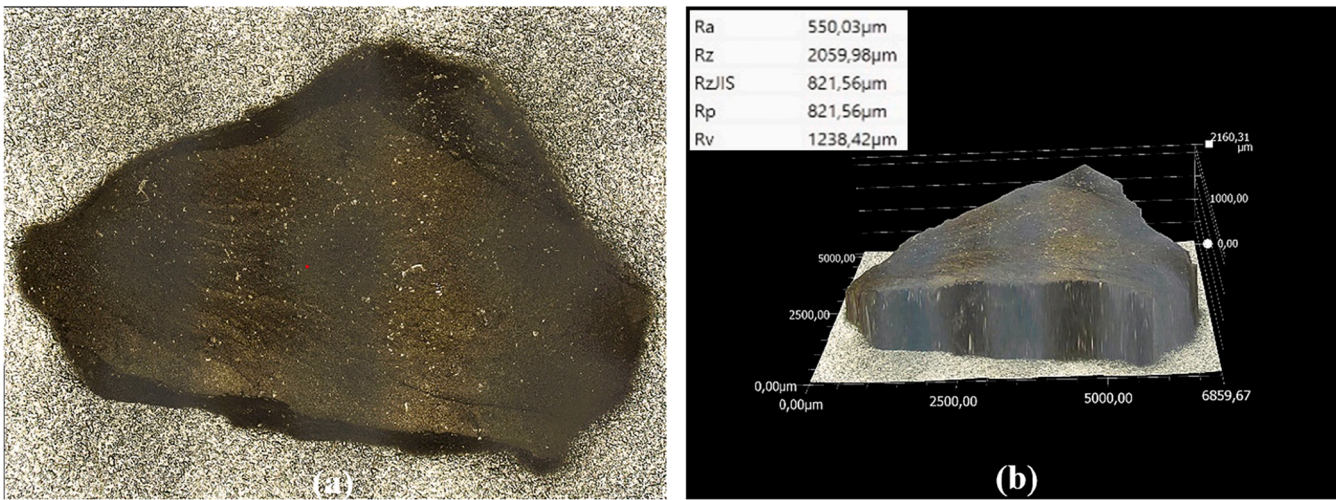


Fig. 5. Three-dimensional surface morphology (a) and roughness profile (b) of untreated rubber aggregate.

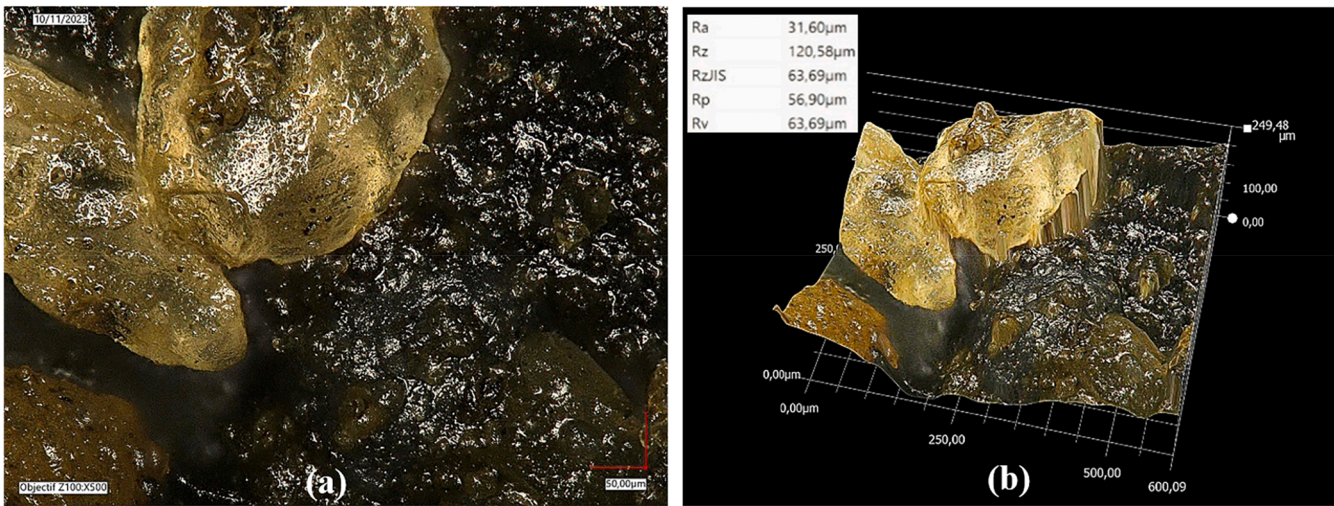


Fig. 6. Three-dimensional surface morphology (a) and roughness profile (b) of hybrid-treated rubber aggregate.

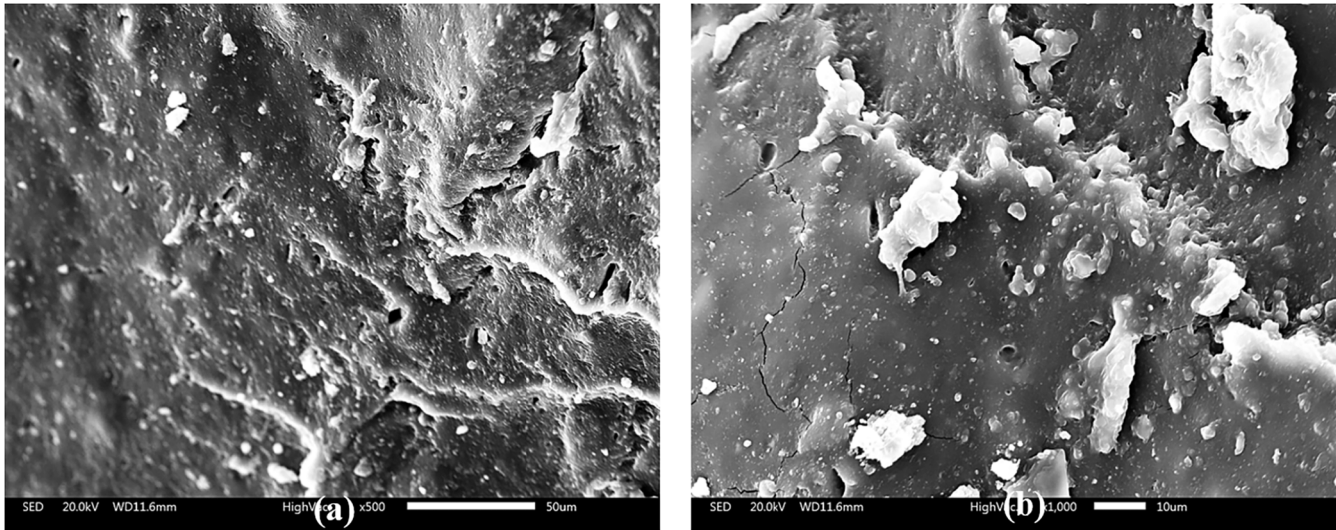


Fig. 7. SEM analysis of untreated rubber aggregates: (a) at 500 × magnification and (b) at 1000 × magnification.

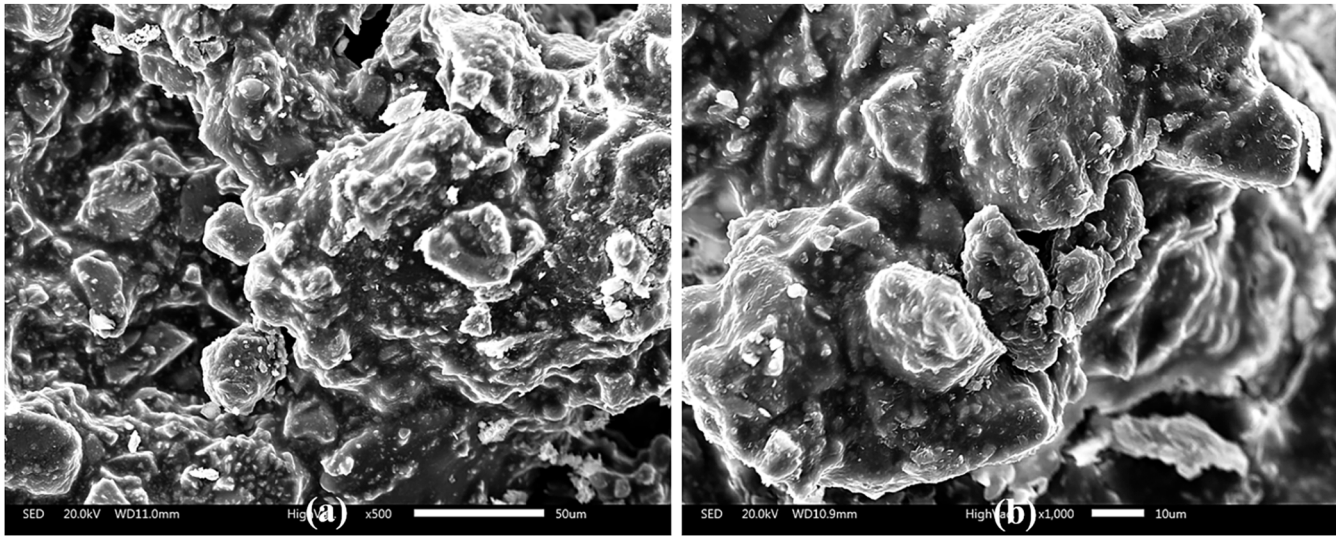


Fig. 8. SEM analysis of hybrid-treated rubber aggregates: (a) at 500 × magnification and (b) at 1000 × magnification.

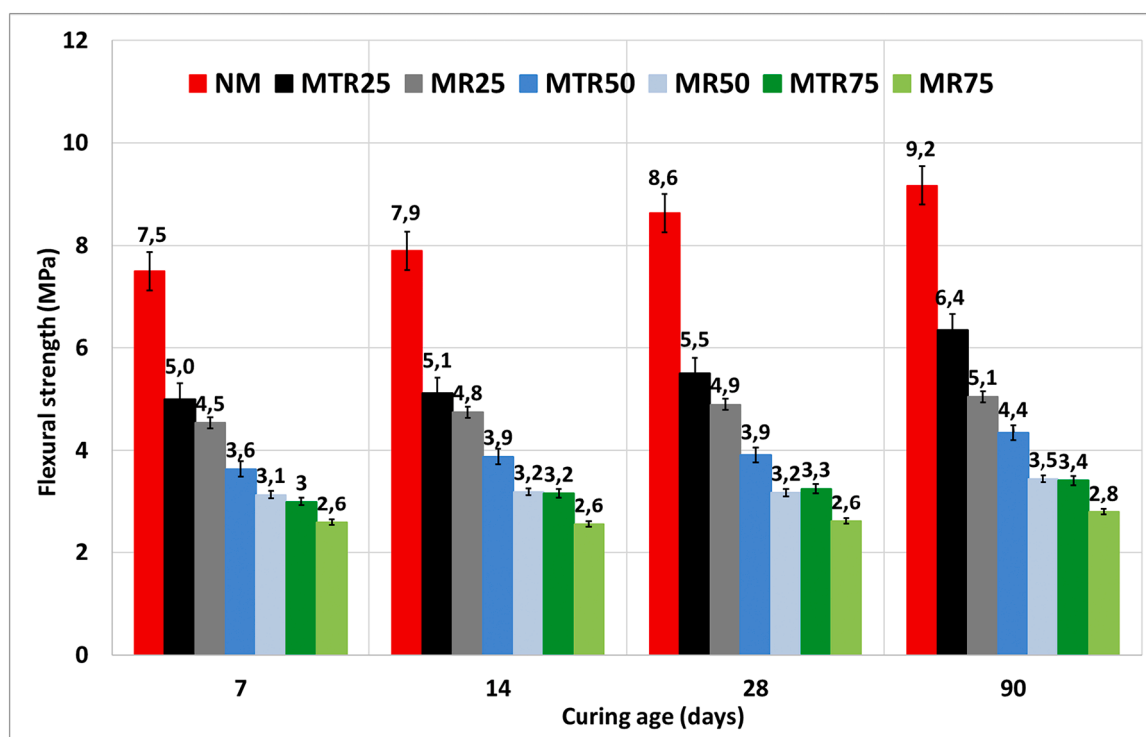


Fig. 9. Flexural Strength Development of Modified Mortars at Different Curing Ages.

with increasing rubber content and persist across all curing ages, are consistent with the findings of Thomas et al. [38] and Khaloo et al. [39], who reported that weak adhesion and deficient stress transfer within the rubber-paste ITZ promote early crack initiation.

Although flexural strength decreased with increasing rubber content, the inclusion of rubber aggregates had a positive influence on the C. Owing to their elastic nature, rubber particles can deform under tensile stress and absorb part of the energy released during crack propagation. When properly bonded to the matrix, these aggregates act as crack arresters, delaying fracture and promoting gradual load redistribution. This behaviour was particularly noticeable in mortars containing hybrid-treated rubber aggregates, where crack propagation was slower and less brittle. It should be noted that this ductile behaviour was not quantified experimentally in the present study, as the EN 196–1 flexural test setup used does not record load-displacement curves. The test is force-controlled and non-instrumented, preventing any quantitative evaluation of fracture energy, post-peak response, or ductility indices for treated and untreated rubber mortars. For this reason, the fracture behaviour discussed here is based solely on consistent visual observations of the failure mode. Despite this limitation, the qualitative trends observed in our study are fully consistent with the literature. Noaman et al. [54] reported that rubberised cementitious composites can exhibit an increase in fracture energy of up to 40–70 % depending on rubber content, even though their flexural strength decreases. This demonstrates that rubber particles may reduce peak tensile resistance while improving crack-bridging capacity and post-cracking energy absorption. Similar tendencies were also noted by Ma et al. [55] and Gesoglu et al. [56] and Kilani et al. [57], who observed reduced brittleness and delayed crack propagation in rubber-modified mortars. The visual observations made in the present work therefore align with previously quantified findings, supporting the interpretation that hybrid-treated rubber aggregates promote a more ductile and progressive fracture mechanism compared with untreated rubber.

Hybrid-treated mortars (MTR series) exhibited significantly higher flexural strength at all curing ages compared with untreated mixes. At 7 days, MTR25, MTR50 and MTR75 reached 5, 3.6 and 3 MPa, representing increases of approximately 11 %, 16 % and 15 % compared with MR25, MR50 and MR75, respectively. At 14 days, MTR25, MTR50 and MTR75 reached 5.1, 3.9 and 3.2 MPa, corresponding to gains of 8 %, 22 % and 23 %. At 28 days, the same mixtures achieved 5.5, 3.9 and 3.3 MPa, representing increases of 12 %, 23 % and 27 %. At 90 days, MTR25, MTR50 and MTR75 reached 6.4, 4.4 and 3.4 MPa, corresponding to gains of 26 %, 26 % and 22 % relative to the untreated mixes. The hybrid treatment therefore provided flexural strength improvements ranging from 11 % to 27 % at early ages and up to 22–26 % at 90 days, depending on curing age and rubber content.

These improvements exceed the typical 6–12 % gains reported for single-mechanism surface treatments such as silane coupling (6–8 %) [45], NaOH-epoxy combinations (\approx 10 %) [46], or mineral coatings (<12 %) [48]. The superior performance of the hybrid treatment results from the synergistic effect of its organic and mineral components: the epoxy resin enhances adhesion and flexibility, while the silica fume and fine sand improve surface roughness and stimulate additional hydration, creating a denser and more cohesive ITZ. Although epoxy resins are hydrophobic, the S&P Resin 55 HP used in this study has a sufficiently low viscosity to wet the micro-reliefs of the rubber surface before curing. As the resin hardens, it immobilises the fine mineral particles and forms a continuous

mineral shell that provides a hydrophilic, rough and mechanically compatible surface, improving bonding within the ITZ as confirmed by SEM and SEM-EDS analyses. The improved interfacial bonding observed microscopically translates directly into enhanced mechanical performance, as the ITZ formed around the treated aggregates not only restores strength but also modifies the fracture mode, promoting ductility and energy absorption under bending loads.

In addition to strength evolution, a statistical analysis was performed using the standard deviation and the coefficient of variation (COV), based on triplicate flexural tests. Error bars appear in Fig. 9, and all numerical values are reported in Table 4. The standardized mortar showed low dispersion at all ages, with COV values between 0.84 % and 2.67 %. The untreated rubber mortars exhibited clearly higher variability, with values reaching 6.93 % for MR25 and 6.48 % for MR50 at 7 days, and increasing further at later ages to 12.20 % for MR25 and 15.72 % for MR75. This behaviour reflects the heterogeneous nature of the rubber-paste interface during early curing and the sensitivity of these mixes to local defects.

The hybrid-treated mortars followed a different trend. Although their COV values were also elevated at 7 days, particularly for MTR25 which reached 14.52 %, they decreased markedly as curing progressed. From 14 days onward, several hybrid-treated formulations showed lower variability than the corresponding untreated mixes at the same rubber content. At 90 days, the COV values of MTR25 (2.73 %), MTR50 (1.22 %) and MTR75 (4.20 %) approached those of the standardized mortar and remained well below the highest values measured for the untreated mixes.

The values reported in Table 4 therefore highlight a clear distinction between the two series. The untreated mortars maintained relatively high variability throughout curing, whereas the hybrid-treated mortars gradually stabilised and achieved, at later ages, levels of reproducibility that were close to those of the reference mortar. The beneficial effect of the hybrid treatment becomes particularly evident beyond 14 days, when the influence of curing age exceeds that of early-age heterogeneities.

The compressive strength results presented in Fig. 10 confirm the beneficial effect of the hybrid surface treatment on the performance of rubberized mortars. The reference mortar reached 38.9 MPa at 7 days, 53.9 MPa at 14 days, 62 MPa at 28 days and 71.3 MPa at 90 days, showing continuous hydration and matrix densification. Each strength value represents the mean of six specimens, ensuring representative and reliable results for all mixtures. Mortars with untreated rubber aggregates showed significant strength losses compared with the reference at all ages. At 7 days, MR25, MR50 and MR75 reached 18.2, 9.8 and 4.3 MPa, corresponding to reductions of approximately 53 %, 75 % and 89 % relative to the reference. At 14 days, MR25, MR50 and MR75 reached 22.0, 11.8 and 5.2 MPa, again reflecting substantial reductions. At 28 days, the reductions were approximately 18 %, 36 % and 55 % for 25 %, 50 % and 75 % replacement levels, respectively, consistent with Li et al. [37] and Siddique and Naik [40]. At 90 days, untreated mortars reached 29.8, 12.1 and 6.2 MPa, maintaining large reductions of 58 %, 80 % and 91 % relative to the reference mortar. These losses result from poor adhesion and the high deformability of untreated rubber, which create a weak and porous ITZ and limit the ability to carry compressive stresses effectively.

In contrast, mortars containing hybrid-treated rubber aggregates exhibited clear strength recovery at all curing ages. At 7 days, MTR25, MTR50 and MTR75 reached 23.4, 12.0 and 6.3 MPa, representing gains of 29 %, 23 % and 46 % compared with MR25, MR50 and MR75. At 14 days, compressive strength increased by 31 %, 33 % and 42 % for MTR25, MTR50 and MTR75, respectively. At 28 days, MTR25, MTR50 and MTR75 reached 31.3, 15.9 and 7.8 MPa, representing gains of 23 %, 54 % and 45 % relative to the untreated mixes. At 90 days, these strengths increased further to 42.1, 21.4 and 10.9 MPa, corresponding to gains of 41 %, 54 % and 76 %, showing sustained and significant improvement over time. Even at 75 % replacement, treated mortars maintained considerably higher strength than untreated ones, demonstrating the stability of the hybrid surface treatment at high rubber contents.

The strength gain between 28 and 90 days highlights the pozzolanic effect of the silica fume contained in the hybrid surface treatment. During this period, amorphous silica reacted with calcium hydroxide, generating additional hydration products that filled microvoids and refined the ITZ. The MTR25 mixture increased from 31.3 MPa at 28 days to 42.1 MPa at 90 days (+34.5 %), while the reference mortar improved by only about 15 % and MR25 showed limited improvement. This late-age improvement is more pronounced in compression than in flexure because compressive strength depends more directly on the compactness and continuity of the ITZ [56,58,46,48]. These compressive strength recoveries exceed those obtained with conventional epoxy-only or silica-based coatings, which typically provide gains of 10–15 % [45–48]. The combined action of the epoxy resin, silica fume and fine sand creates a cohesive hybrid layer that strengthens interfacial bonding and improves stress distribution: fine sand particles act as micro-anchors, while the epoxy resin enhances mechanical compatibility and contributes to more uniform load transfer across the matrix.

The compressive strength and COV values presented in Table 5 allow for an comprehensive assessment of the mechanical behaviour of the different mixtures. The coefficient of variation (COV), defined as the ratio of the standard deviation to the mean value ($COV = SD / \text{mean} \times 100$), was calculated for each curing age to quantify the dispersion of the measured strengths. The reference mortar

Table 4
Flexural strength (MPa) and COV (%) of mortars at different curing ages.

Mix	7 days (MPa / COV %)	14 days (MPa / COV %)	28 days (MPa / COV %)	90 days (MPa / COV %)
NM	7.5 / 2.67	7.9 / 0.84	8.6 / 1.46	9.2 / 2.08
MR25	4.5 / 6.93	4.8 / 1.77	4.9 / 7.7	5.1 / 12.2
MR50	3.1 / 6.48	3.20 / 5.54	3.2 / 7.3	3.5 / 3.83
MR75	2.6 / 2.79	2.6 / 1.96	2.6 / 3.87	2.8 / 15.72
MTR25	5 / 14.52	5.1 / 4.89	5.5 / 1.82	6.4 / 2.73
MTR50	3.6 / 2.77	3.9 / 3.55	3.9 / 5.04	4.4 / 1.22
MTR75	3 / 6.15	3.2 / 11.46	3.3 / 4.23	3.4 / 4.2

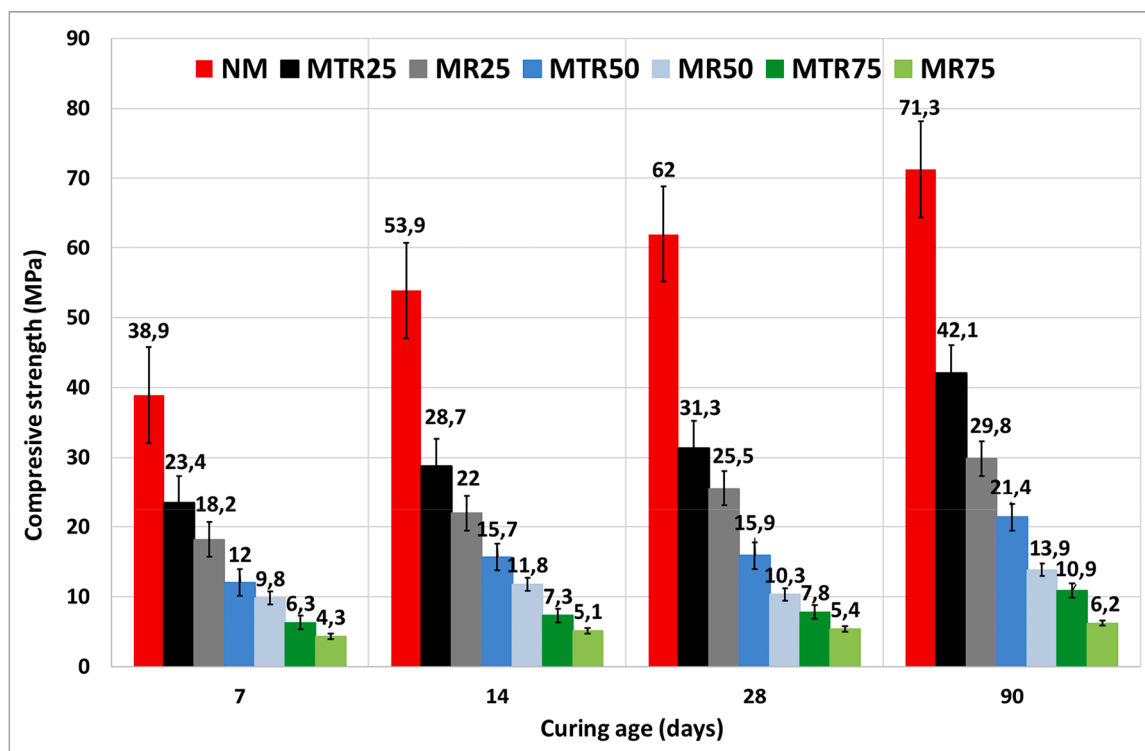


Fig. 10. Compressive Strength Development of Modified Mortars at Different Curing Ages.

Table 5

Compressive strength (MPa) and COV (%) of mortars at different curing ages.

Mix	7 days (MPa / COV %)	14 days (MPa / COV %)	28 days (MPa / COV %)	90 days (MPa / COV %)
NM	38.9 / 5.26	53.9 / 6.15	62.0 / 4.02	71.3 / 5.31
MR25	18.2 / 3.92	22 / 5.55	25.5 / 4.82	29.8 / 4.90
MR50	9.8 / 12.07	11.8 / 5.91	10.3 / 3.88	13.9 / 5.77
MR75	4.3 / 5.49	5.2 / 3.53	5.4 / 5.88	6.2 / 5.91
MTR25	23.4 / 1.56	28.8 / 3.61	31.3 / 2.62	42.1 / 4.55
MTR50	12.0 / 5.85	15.7 / 10.73	15.9 / 3.63	21.4 / 3.63
MTR75	6.3 / 3.23	7.3 / 14.41	7.8 / 11.94	10.9 / 5.86

combined high compressive strength with moderate variability, confirming the stability of the standardized matrix. The untreated rubber mortars showed both reduced strength and higher dispersion, particularly at early ages, where strength losses were accompanied by COV values exceeding 10 % for some formulations. The hybrid-treated mortars displayed a different trend: although their strength remained lower than that of the reference at equal curing age, the treated mixes achieved noticeably higher strength than their untreated counterparts while also exhibiting reduced variability after 14 days. At 90 days, the COV values of MTR25, MTR50 and MTR75 were comparable to those of the reference mortar, and their strengths increased to 42.1, 21.4 and 10.9 MPa, respectively. These combined observations show that the hybrid surface treatment improves both the compressive performance and the reproducibility of rubberized mortars once sufficient curing has been reached.

The microstructural observations provide direct evidence of the mechanisms underlying these mechanical trends. Following aggregate characterization, the microstructure of the hardened rubberized mortars was examined to evaluate how the hybrid surface treatment influences the ITZ and the integrity of the matrix (Figs. 11–12). In the untreated mixture (Fig. 11), the ITZ appears porous and discontinuous, characterized by microcracks and gaps separating the rubber surface from the surrounding paste. The weak adhesion results from the smooth, non-polar nature of the rubber, which restricts the formation of calcium silicate hydrate (C-S-H) phases and hinders proper bonding. Using the 10 μm scale bar as reference, the apparent ITZ width in the untreated mortar is estimated at about 10–14 μm along representative sections, acknowledging that this value is semi-quantitative due to edge effects and surface relief. Such a wide and defective ITZ is fully consistent with the severe flexural and compressive strength losses and with the brittle fracture observed in MR mixes.

In contrast, the treated mortars (Fig. 12) exhibit a compact and continuous ITZ with fewer voids and a denser structure. The Hybrid

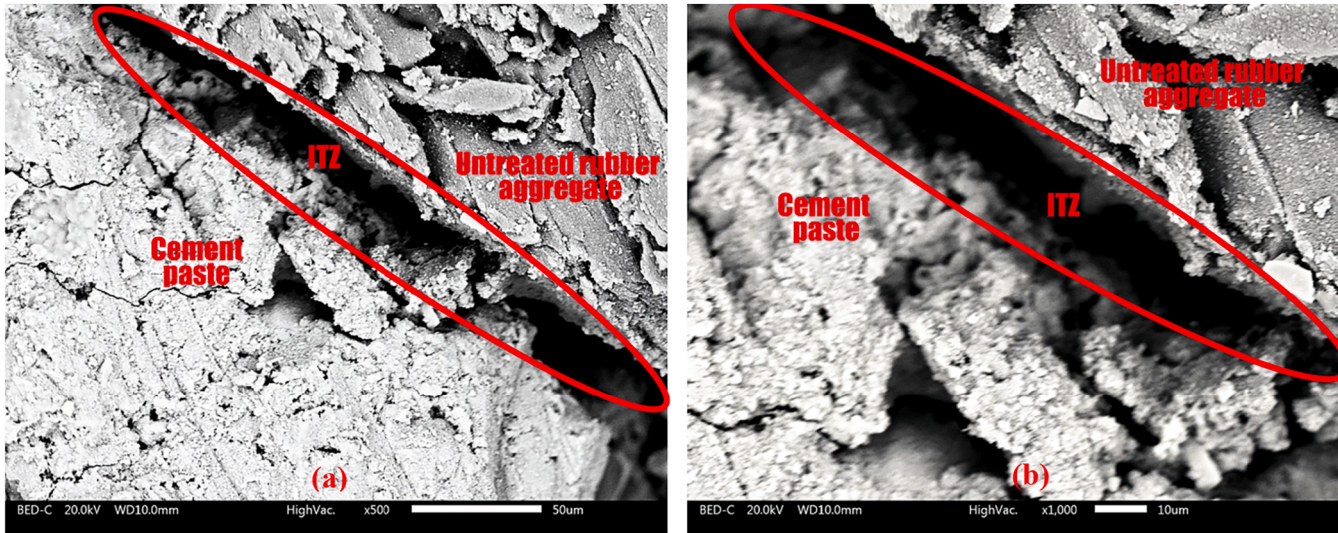


Fig. 11. SEM images of mortar incorporating untreated tire rubber at Magnifications of $\times 500$ (a) and $\times 1000$ (b).

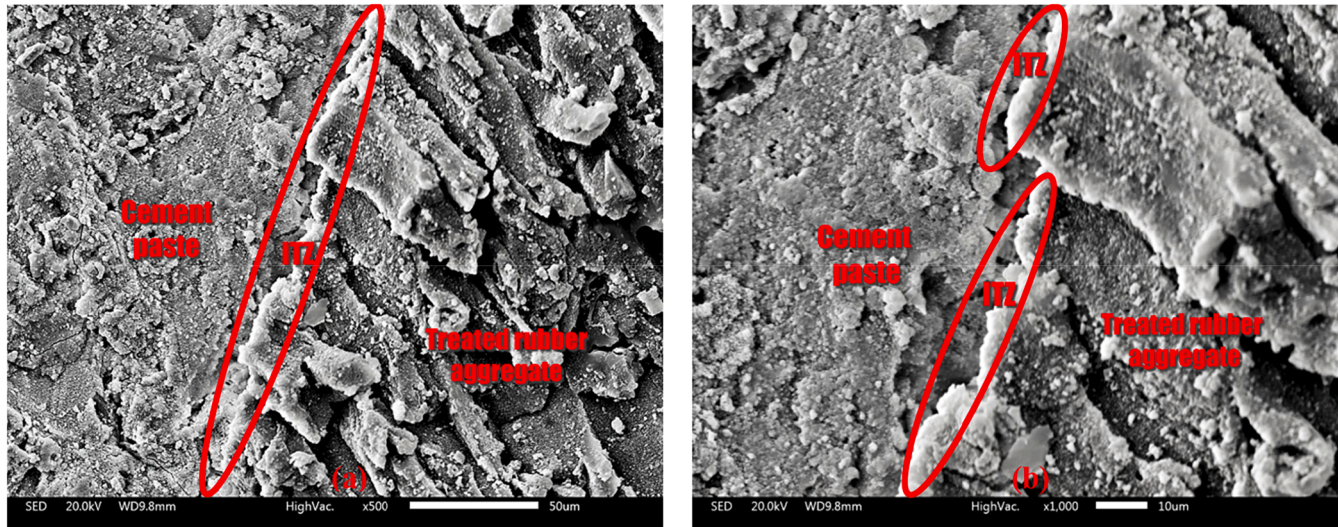


Fig. 12. SEM Images of Mortar Containing Treated Tire Rubber at Magnifications of $\times 500$ (a) and $\times 1000$ (b).

surface treatment, composed of epoxy, cement, silica fume and fine sand, creates a rough and chemically compatible interface that enhances both mechanical interlocking and hydration activity. Compared with the untreated mix, where the ITZ appeared wide ($\approx 14\text{--}15\ \mu\text{m}$) and loosely connected, the treated mortars exhibit a continuous and compact interfacial layer only $1\text{--}2\ \mu\text{m}$ thick. The resulting interface shows fewer microvoids and a more homogeneous bond with the surrounding paste. This transformation from a weak, porous boundary into a dense and cohesive layer explains both the partial recovery of flexural strength and the significant compressive strength gains observed in the MTR series, as the refined ITZ more effectively transfers stresses and delays crack initiation around rubber particles.

To complement these morphological observations, a coupled SEM–EDS analysis was conducted to determine the chemical composition and phase distribution across three representative regions of the hybrid-treated system: the cement paste, the treated rubber aggregate and the ITZ. In each case, the red cross marks the exact location where the EDS acquisition was performed.

The spectrum associated with Fig. 13, corresponding to the cement paste, displays dominant peaks of calcium (Ca), silicon (Si) and oxygen (O), together with minor aluminium (Al) and iron (Fe). This composition is characteristic of hydrated cementitious phases rich in C-S-H and portlandite (CH) and serves as a chemical reference for comparison with the coated rubber and the transition zone.

The spectrum shown in Fig. 14, recorded on the surface of a hybrid-treated rubber aggregate, exhibits a more complex composition arising from the hybrid coating. Strong Ca-K, Si-K and O-K peaks confirm the presence of hydration products adhering to the coated surface. Silicon peaks not directly associated with calcium indicate the presence of silica fume, partly involved in pozzolanic reactions with portlandite while some unreacted particles remain detectable. The Zn-K and S-K peaks reflect the vulcanised rubber substrate beneath the coating. Additional low-intensity Si-O signals with minimal calcium correspond to siliceous sand grains embedded within the mineral layer and contributing to mechanical interlocking.

The spectrum presented in Fig. 15, corresponding to the ITZ, reveals a balanced mixture of cementitious and siliceous phases. Strong Ca, Si and O peaks indicate a dense concentration of C-S-H gels and portlandite, while moderate Al and S contents point to the presence of ettringite and AFm-type hydrates. The detection of Zn confirms the immediate proximity of the rubber aggregate. The overall chemical continuity observed in the ITZ shows that hydration and pozzolanic products have progressively filled microvoids and sealed the transition zone.

Similar chemical continuity has been reported by Najim and Hall [58], who showed that hybrid mineral coatings promote mechanical interlocking and continuous hydration across the rubber–cement interface. In the present work, the SEM–EDS analysis confirms that the hybrid treatment produces a chemically integrated and mechanically anchored interface. The combined action of silica fume, fine sand and the epoxy-cement coating enhances both physical interlocking and chemical bonding, forming a cohesive ITZ capable of efficient stress transfer under bending and compression.

These microstructural and chemical observations are consistent with the findings of Chou et al. [59], who reported that chemical modification increases rubber surface polarity and strengthens long-term interfacial bonding, and with Gesoglu et al. [56], who showed that silica fume addition refines the ITZ and reduces microcracking by promoting secondary C-S-H formation. Overall, the hybrid treatment clearly refines the ITZ, reduces porosity and improves interfacial continuity, which explains the enhanced mechanical response of the rubberised mortars.

3.3. Cost-performance evaluation of the hybrid treatment

A detailed cost-performance assessment was conducted to evaluate the economic relevance of the hybrid mineral-epoxy treatment developed in this study. Treating 20 kg of rubber aggregates requires 6 kg of a mineral blend composed of 50 % sand, 40 % CEM I

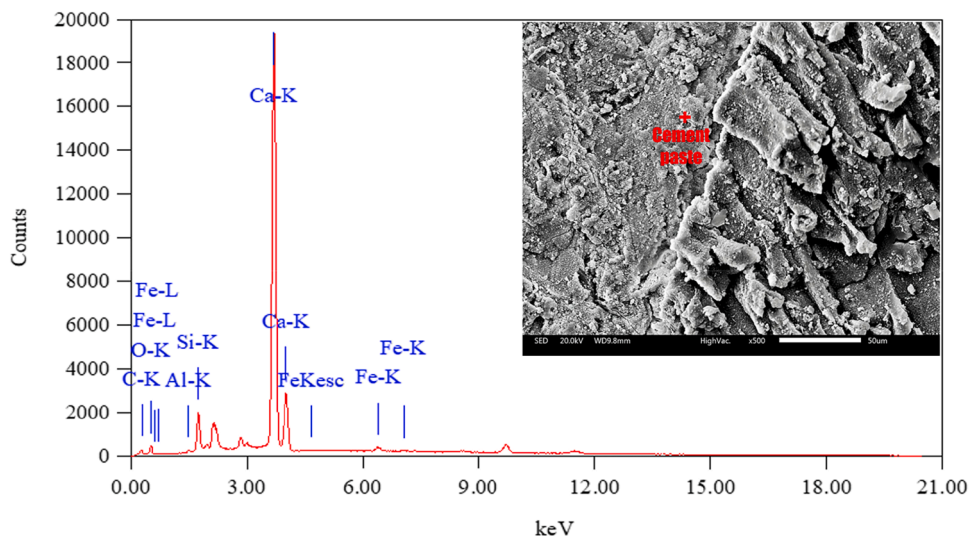


Fig. 13. EDS Spectrum at the Cement Paste.

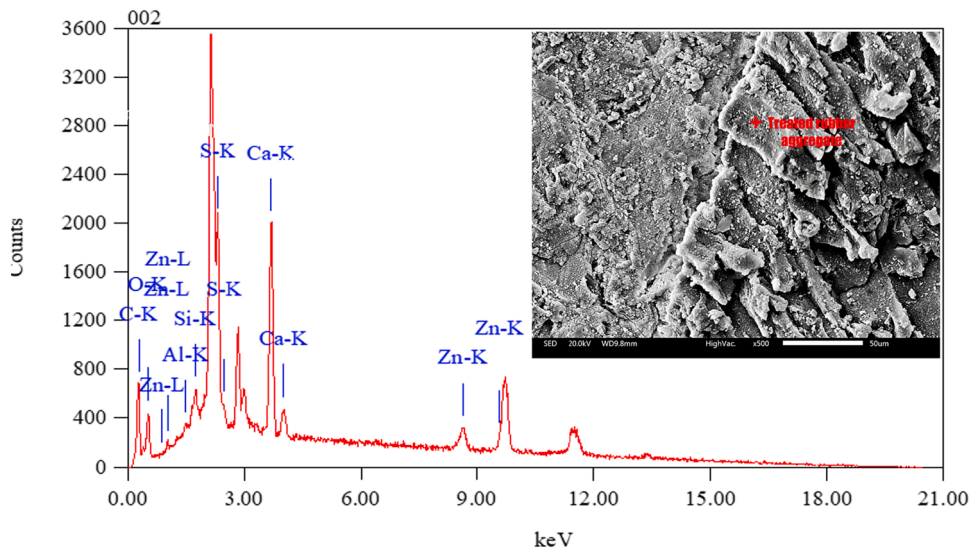


Fig. 14. EDS Spectrum at the treated rubber aggregate.

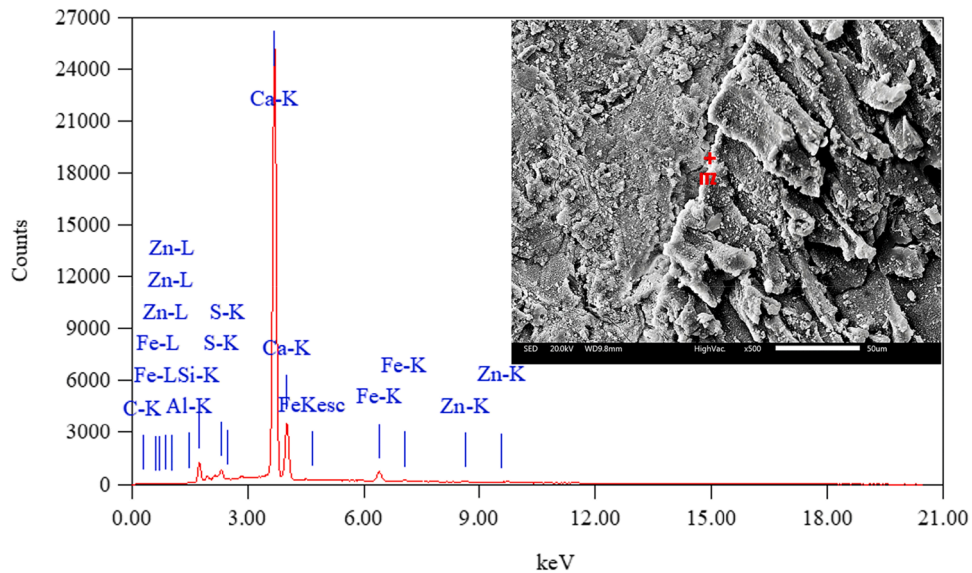


Fig. 15. EDS Spectrum at the Rubber-Cement Interface (ITZ).

52.5 N cement and 10 % silica fume, together with 1.5 L of epoxy resin. The cost of each constituent was determined from the pricing provided by the corresponding manufacturers: CEM I 52.5 N at approximately 600 €/t (Lafarge), S&P Resin 55 HP at 112 € for 6 L (S&P Reinforcement) and untreated rubber aggregates at 180 €/t (Delta Gom). Based on these values, the quantities of sand, cement and silica fume required for 20 kg of rubber aggregates cost 0.06 €, 3.60 € and 2.40 €, while the resin represents the main expense, with 1.5 L costing about 28 €. The total cost of the hybrid treatment therefore amounts to approximately 34 € for 20 kg of rubber aggregates, corresponding to an added cost of about 1 700 €/t. Including the cost of untreated rubber aggregates, the total cost of hybrid-treated aggregates reaches approximately 1 900 €/t.

The resin dosage used in this study ensured complete and uniform coating, producing an adhesive film of about 25 μm . Since such a thickness is not required for the adhesive function of the resin, the film can be reduced without affecting the anchorage of the mineral skeleton. Reducing the resin layer from 25 μm to approximately 10 μm corresponds to a 60 % decrease in resin consumption (from 1500 g to about 600 g). As the resin represents the dominant share of the treatment cost, this optimisation would reduce the treatment cost by the same proportion, decreasing it from about 1 700 €/t to approximately 680 €/t. In addition to the economic benefit, decreasing the thickness of the polymer film increases the proportion of mineral grains directly exposed to the cementitious environment, where they effectively participate in cement hydration reactions and pozzolanic reactions with silica fume, instead of

remaining fully embedded within the resin. This enhances the chemical reactivity of the mineral phase and strengthens the contribution of the mineral skeleton to the interfacial zone.

A comparison with the surface modification methods evaluated by Assaggaf et al. [60] clarifies the economic position of the hybrid coating. Once converted into euros, the cost of rubber aggregates increases from approximately 258 €/t for untreated particles to 331 €/t for NaOH-treated particles, corresponding to an increase of 28 % relative to untreated rubber, and to 655 €/t for KMnO₄-treated particles, corresponding to an increase of 154 %. Cement coating is the only treatment that decreases production cost, reducing it to around 201 €/t, which represents a reduction of 22 % compared with untreated rubber. However, the mechanical effectiveness of the cement coating is restricted to substitution levels that do not exceed 40 %, and the NaOH and KMnO₄ treatments lose efficiency beyond 10–20 % substitution because of particle agglomeration.

When evaluated at comparable substitution levels, the hybrid mineral-epoxy treatment provides a much more favourable cost-performance balance. Flexural strength increased by 6–27 %, and compressive strength improved by 23–41 % at 25 % rubber, 22–54 % at 50 % and 40–76 % at 75 %. These gains are substantially higher than those reported for cement-coated or chemically treated rubber, particularly at medium and high substitution levels where the latter methods rapidly lose effectiveness. The hybrid coating also remains effective up to 75 % rubber substitution, which ensures a more advantageous cost-to-performance ratio.

Although its initial material cost is higher than that of the treatments previously reported, the stable and substantial mechanical improvements observed at all substitution levels confirm the technical and economic relevance of the hybrid mineral-epoxy treatment. The development of a dense and continuous interfacial layer, the robust mechanical anchorage provided by the coating and the potential reduction of resin consumption through optimisation of the adhesive film thickness together reinforce the practical feasibility of this surface treatment for improving the behaviour of rubberised mortars.

3.4. Environmental and sustainability benefits

The hybrid mineral-epoxy treatment provides several environmental advantages. By enabling reliable mechanical performance at medium and high rubber substitution levels, it supports the reuse of end-of-life tire particles and reduces the extraction of natural sand, thereby limiting the environmental impacts associated with quarrying and landfill disposal. The incorporation of silica fume in the coating also contributes to sustainability by lowering clinker demand and promoting pozzolanic reactions that densify the interfacial zone and enhance long-term durability.

Optimising the resin content further decreases the environmental footprint linked to polymer production while increasing the proportion of mineral grains that actively participate in hydration. In addition, unlike chemical pre-treatments based on NaOH or KMnO₄ solutions, which rely on aggressive reagents and generate hazardous liquid wastes, the hybrid treatment avoids the use of corrosive chemicals and eliminates washing steps, reducing both environmental risk and operator exposure. These aspects are consistent with recent sustainability-oriented studies on rubber pre-treatments [12,19].

3.5. Limitations and future recommendations

Despite the clear mechanical improvements achieved with the hybrid treatment, several limitations should be considered. The economic analysis presented here reflects current french market conditions, and variations in material availability, transportation costs and regional pricing may influence the applicability of the treatment in other contexts. A broader economic evaluation would therefore provide a more comprehensive understanding of its feasibility.

The mechanical characterisation conducted in this study focused on compressive and flexural strengths supported by SEM observations that confirmed the formation of a denser and more coherent ITZ around the treated rubber particles. While these results provide a solid qualitative and quantitative basis for interpreting the enhanced mechanical behaviour, the present work does not include stress-strain curves or a detailed analysis of fracture mechanisms. strength values alone do not fully describe post-peak behaviour or deformation capacity, particularly for rubberised composites where ductility is a key performance aspect.

Future research should therefore incorporate complete stress-strain curves obtained from both compression and flexural tests performed on concrete incorporating hybrid-treated rubber aggregates. Flexural testing is especially relevant for quantifying the ductility contribution of the Hybrid surface coating, including energy absorption, crack-bridging capacity and post-peak deformation. Such measurements would provide quantitative and visual evidence of fracture and deformation mechanisms and clarify how the Hybrid surface coating influences crack initiation, propagation and overall deformability. They would also enable more rigorous comparisons with other surface treatments and reinforce the mechanical justification for adopting the hybrid method.

Additional investigations using complementary characterisation techniques such as nanoindentation could offer deeper insight into the microscale interaction between the coating and the cement paste. Further optimisation of the mineral-epoxy formulation, including adjustments in coating thickness, mineral composition or resin dosage, may also help reduce cost while maintaining or enhancing performance. Collectively, these developments would refine the understanding of the Hybrid surface coating's mechanical contribution and strengthen its potential for large-scale implementation in rubberised concrete.

4. Conclusions

This study examined how a single-step hybrid surface treatment applied to tire-derived rubber aggregates can improve the behaviour of cement mortars. By combining epoxy resin, cement, silica fume, and fine sand within one formulation, the coating enhanced both the microstructure and the overall performance of rubberized mortars. Microscopic observations confirmed the

formation of a compact and continuous interfacial transition zone (ITZ), demonstrating improved interaction between the coated rubber particles and the surrounding cement paste. These results highlight the relevance of the hybrid treatment as an efficient approach for incorporating end-of-life tire aggregates into cement-based materials.

The main outcomes of this research can be summarised as follows:

1. **Hybrid treatment mechanism:** The combined epoxy–mineral coating provides complementary adhesion and mechanical anchorage. The epoxy film ensures initial wetting of the rubber surface, while the mineral skeleton of sand, cement and silica fume forms a reactive and textured outer layer, enabling the aggregates to contribute to load transfer rather than act as inert inclusions.
2. **Mechanical performance:** Mortars incorporating hybrid-treated rubber aggregates recovered a large proportion of the strength typically lost with untreated rubber. Flexural strength increased by 11–27 %, and compressive strength by 23–76 %, depending on substitution level and curing age. These gains exceed those generally reported for conventional single-step treatments such as NaOH, KMnO₄ or cement coatings, particularly at medium and high substitution levels.
3. **Microstructural improvement and its contribution to strength recovery:** SEM–EDS analyses showed that the hybrid treatment produces a thinner, denser and more continuous ITZ. The mineral skeleton enhances roughness and mechanical interlocking, while silica fume contributes to the formation of secondary C-S-H, further strengthening the interface. This microstructural refinement directly supports and explains the improved mechanical behaviour.
4. **Time-dependent effects:** Strength gains became more pronounced between 28 and 90 days, reflecting progressive ITZ densification driven by pozzolanic reactions. These long-term improvements suggest favourable durability prospects for hybrid-treated systems.
5. **Cost–performance and sustainability relevance:** The hybrid coating provides a favourable balance between cost and mechanical efficiency, particularly when resin consumption is optimised. By enabling high rubber substitution levels, reducing the consumption of natural sand and avoiding the use of corrosive chemical pre-treatments, the method strengthens both the economic and environmental relevance of rubber recycling in cementitious materials.

The results obtained at the mortar scale indicate that hybrid-treated rubber aggregates can be effectively incorporated into concrete. Ongoing investigations address their application in FRP-confined reinforced concrete columns under axial compression. The outcomes of this work, currently in progress, are expected to confirm the potential of the hybrid treatment for more durable and sustainable structural applications.

CRedit authorship contribution statement

Achouak Yousfi: Writing – review & editing, Writing – original draft, Resources, Investigation, Conceptualization. **MESBAH Habib Abdelhak:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Resources, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization. **Mohamed Saadi:** Writing – review & editing, Investigation.

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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Data availability

No data was used for the research described in the article.

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