Article

Performance Evaluation of Cementitious Composites Containing Granulated Rubber Wastes, Silica Fume, and Blast Furnace Slag

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Abstract: In this study, rubberized cementitious materials are produced with recycled rubber waste as an alternative to fine aggregate. Mixtures with different additions to rubber wastes (RW), silica fume (SF), and blast furnace slag (BFS) have been designed and characterized. Hardened properties including compressive and bond strength, shrinkage, water-accessible porosity, rapid chloride migration, and microstructure were investigated. The results show that the addition of SF and BFS improves the performances of rubberized mortars and reduces shrinkage. The incorporation of 5% RW with 20% BFS increases compressive strength and reduces water-accessible porosity. Ion chloride resistance was enhanced by a combination of 15% RW, 8% SF, and 20% BFS. The addition of SF and BFS as cement replacement improves the performance of mortars due to their filling effect and a pozzolanic reaction, which has been verified by a microstructural analysis.

Keywords: rubber waste; rubberized mortar; mechanical characteristics; pull-out test; bond strength; chloride mitigation

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1. Introduction

Energy conservation and reduction of carbon emissions have become critical in the world [1], and it is well known that 8 to 10% of global CO₂ emissions are produced by the cement industry, which is seen as one of the leading causes of climate change [2]. Concrete typically contains approximately 12% cemented materials, 8% water, and 80% aggregates. The extraction, processing, and transport of vast quantities of aggregates, together with raw materials required each year for cement production, consumes a considerable amount of energy and harms the ecology of the planet [3]. Faced with the growing demand for material resources and the need to preserve the environment, it is necessary to explore and study possibilities for the re-use and recovery of waste and industrial by-products, especially in the field of civil engineering [4].

Used tires are non-hazardous waste, but their storage presents a risk to the environment and humanity if there is no additional fire prevention at the storage site; this creates a significant waste management problem. The rubber industry is one of many sources of waste and produces above 1.5 billion new tires worldwide each year [5]. For example, in Europe, some 3.4 million tons of old tires are disposed of each year [6]. In India, 112 million discarded tires are produced annually [7] and in 2018, around 14.58 million tons of used tires have been produced in China [8]. Low bulk density, drainage capacity, compressibility and thermal conductivity of tire waste can be used in geotechnical and civil engineering applications such as light backfills, prevention of frost-heating, landfill drainage, road and highway pavement asphalt, sports surfacing, and filling materials for turf grass [9–12].

Several studies, including the incorporation of rubber aggregates into cement matrices, have found rubber aggregates to be viable in substitution of sand and gravel in concretes and mortars. Thomas et al. [13] studied the performances of high-strength rubber concrete containing rubber from used tires (size from 0% to 20% in multiples of 2.5%); it was found that water absorption and abrasion resistance were better than that of the reference concrete, while depth of water penetration, pull-off, flexural tensile, and compressive strength of rubber concrete were less than that of the reference mix. Ganjian et al. [14] also investigated the performances of concrete mixes incorporating 5%, 7.5%, and 10% of discarded tire rubber as aggregate and cement replacements and the results showed a 28-day compressive strength reduction—about 10 to 23% and 20 to 40%—and a flexural strength of about 37% and 29% for aggregates and for the cement replacement, respectively. Valente and Sibai [15] conducted a review in which various amounts of waste tire powder were combined with cement concrete mixtures to produce a final product with mechanical properties suitable for engineering applications. It was concluded that a good compressive strength can be obtained by replacing 30% of the powdered tire with crushed sand. Compressive strength decreases as the percentage of aggregation between crumb rubber and crushed sand increases. Aggregation replacement of crumb rubber and crushed sand results in a density reduction of around 10%. The modulus of elasticity is proportional to the percentage of rubber added: the more rubber added to concrete, the less elastic the product. Furthermore, less tough concrete means greater strength. The addition of rubber to concrete, on the other hand, increases its toughness. Sambucci et al. [16] conducted a preliminary physical-mechanical analysis on environmentally friendly mortars made of recycled rubber aggregates derived from end-of-life tires that are compatible with the extrusion-based printing process. To partially/ completely replace the sand, 50% and 100% rubber powder (size 0–1 mm), 50% and 75% rubber granules (size 2–4 mm) were used. The results show that the rubber aggregates increase the fluidity and ductility of the mixture, promoting better inter-layer adhesion than the neat mix, resulting in greater mechanical isotropy but decreasing mechanical strength. The incorporation of rubber granules from used tires into cementitious materials is detrimental to compressive strength and tensile strength [17–20]. In return, the composite obtained has a greater ductility and strain capacity [21–27]. Grdić et al. [26] reported an increase in the concrete ductility in the range of 25%, 81.25%, and 93.75%, by partially replacing natural sand with crumb rubber (size 4–0.5 mm) at levels of 10%, 20%, and 30%, respectively, by volume. Huang et al. [27] reported that the increase in the tensile strain capacity was 11.11%, 16.67%, 44.44%, and 66.67% with the inclusion of 10%, 20%, 30%, and 40% rubber sand, respectively.

Previous studies have mentioned that immersing the waste rubber into a solution of NaOH [28], covering it with a thin layer of cement [29], using styrene-butadiene rubber (SBR) [30], using organic sulfur [31], and using a silane coupling agent with a coating [32] all contributed to improved strength. Improved properties were also shown in rubber concrete with addition of silica fume (SF) [33,34], fly ash [35,36], calcareous [37], steel fibers [38], and polyvinyl alcohol fibers (PVA) [39–42]. Copetti et al. [34] replaced Portland cement (7.5% and 15% ratios) with silica fume in rubber concrete, up to 80% gains in compressive strength 28 days with 30% rubber replacement were resulted. The properties of self-compacting rubberized concrete incorporating polypropylene (PP) and steel fibers were studied by Aslani et al. [38]. As a replacement for 20% of fine aggregates and 0.1%, 0.15%, 0.2%, 0.25%, 0.25% of PP fibers and 0.25%, 0.5%, 0.75%, 1% for steel fibers, crumb rubber with size of 2 to 5 mm was used. With the increase in the content of steel fibers, the results show a minor increase in compressive and tensile strengths. The behavior of concrete incorporating crumb rubber as a partial replacement of fine aggregate and polyvinyl alcohol fibers as cement addition was studied by Sadiq et al. [39]. 1% and 2% of PVA (by mass of cement), 5% and 10% of crumb rubber into concrete (by weight of fine aggregate) were incorporated and a positive impact on the engineering properties of concrete was shown by PVA.

Although RW-incorporated cementitious materials were designed and some performances, especially mechanical properties, were identified, it is still necessary to systematically build relations between key design parameters and micro-macro properties. This study aimed to investigate the effect of mineral additives such as silica fume and blast furnace slag, on the physical-mechanical, durability, and microstructural properties of rubberized mortar. Where fine aggregate was replaced with rubber aggregate waste, cement was replaced by BFS and SF. Compressive and bond strength, shrinkage, water-accessible porosity, Rapid migration of chloride, SEM and XRD analysis were assessed and discussed.

2. Materials and Test Procedures

2.1. Materials

The cement used for all mortar mixtures is the OPC class 42.5 produced by Huaxin Cement in China. Its specific surface area and absolute density were $365 \text{ cm}^2/\text{g}$ and 2.56 g/cm^3 , respectively. Fine aggregates with particle (size $\leq 5 \text{ mm}$), absolute and apparent densities of 2.56 and 1.49 g/cm^3 , respectively, were used as well as commercial SF and BFS. In the volume substitution of sand, the RW aggregates resulting from the mechanical grinding of old tires are used. These aggregates have a maximum size of 5.0 mm ; their absolute and apparent densities are 1.21 and 0.44 g/cm^3 , respectively. Table 1 lists the chemical composition of OPC, SF, and BFS. For each mixture, the superplasticizer (polycarboxylic acid water reducing agent) was used in different percentages by weight of the binder to ensure the same workability with varying rubber particle proportions. At 0.1% by weight of the total binder, the stabilizer, Hydroxy Propyl Methyl Cellulose (HPMC), was used to avoid segregation of rubber particles because of the significant difference between their specific density and density of the sand. In order to improve the studied mortar matrix bonding, a synthetic aqueous dispersion resin (SIKALATEX) was used at a fixed cement weight of 7% . 1 kg/L is its density. For the treatment of the surface of rubber waste, sodium hydroxide (NaOH) was used to improve adhesion between rubber waste and cement. The treatment was carried out by immersing the RW in an alkaline solution made by dissolving 10 g of NaOH pastilles in 90 cm^3 of distillate water, resulting in a 10% concentration. The RW was immersed in this saturated aqueous solution for $20\text{--}30$ minutes while agitating the mixture on a regular basis. The removal of excess NaOH adsorption on the surface of the RW required rinsing with distilled water. To ensure that all of the NaOH from the RW was removed, the pH of the solution was tested with a pH paper until it was equal to 7 . Finally, the RW was dried in open air before use. The raw materials used and described earlier in this work are presented in Figure 1.

Table 1. Chemical composition of the OPC, SF, and BFS used (%).

	OPC	SF	BFS		OPC	SF	BFS
Oxydes		(%)		Oxydes		(%)	
CO ₂	-	3.263	-1.621	MnO	-	0.091	0.091
Na ₂ O	-	1.000	0.326	ZnO	-	0.009	-
MgO	3.56	5.954	7.976	SeO ₂	-	0.005	-
Al ₂ O ₃	4.89	10.134	15.495	Br	-	0.002	-
SiO ₂	20.93	23.941	32.823	Rb ₂ O	-	0.004	0.003
P ₂ O ₅	-	0.034	0.018	SrO	-	0.110	0.125
SO ₃	2.27	13.473	2.494	Y ₂ O ₃	-	0.008	0.010
Cl	-	0.040	-	ZrO ₂	-	0.040	0.051
K ₂ O	0.56	0.727	0.478	BaO	-	0.087	0.337
CaO	60.42	38.677	38.853	CeO ₂	-	0.054	-
TiO ₂	-	0.554	0.649				
Fe ₂ O ₃	2.89	1.796	0.273				
Loss on ignition	3.60	-	-

Figure 1. Raw materials used.

2.2. Mixture Proportions and Sample Preparation

In the laboratory, nine mortar mixtures containing the above-mentioned materials were produced, as shown in Table 2, where all the mixtures had a fixed water/binder ratio of 0.45. Different percentages, by weight of the total binder, were used for the superplasticizer introduced in the blends. SF, BFS, and RW contents were used. By mass substitution of the cement, the SF content tested was 8% and the BFS content was 20%. In addition, the RW contents tested were 5%, 10%, and 15% by volume substitution of sand.

Table 2. The proportions of the mortar mixture used (kg/m³).

Mortar Mixture	OPC	Fine Aggregates	Water	RW	BFS	SF	Stabilizer	SP-Plast	Resinous Latex
Ref	450	1350	202.5	0	0	0	0	9.95	0
5RW	450	1282.5	202.5	31.91	0	0	0.45	5.22	31.5
10RW	450	1215	202.5	63.81	0	0	0.45	4.82	31.5
15RW	450	1147.5	202.5	95.72	0	0	0.45	5.49	31.5
5RW+8SF	414	1282.5	202.5	31.91	0	36	0.45	7.38	31.5
5RW+20BFS	360	1282.5	202.5	31.91	90	0	0.45	3.96	31.5
10RW+8SF	414	1215	202.5	63.81	0	36	0.45	6.12	31.5
10RW+20BFS	360	1215	202.5	63.81	90	0	0.45	3.92	31.5
15RW+8SF+20BFS	324	1147.5	202.5	95.72	90	36	0.45	6.53	31.5

Mortar samples were prepared using laboratory mixers. At slow speed, sand, cement, RW, BFS, and SF were blended for 3 min. 80% water, the superplasticizer, the stabilizer and the resinous latex were added once the mixture was homogeneous and mixed at slow speed for 2 minutes. After that, the remaining 20% of water was added and the mixtures were mixed for more than 3 minutes at rapid speed. Tests on the shaking table were carried out immediately to obtain plastic mixtures (workability comprise between 14–18 cm). After that, for each step, fresh mortar was poured into different plastic molds in 2 stages and vibrated for 30 s, then sealed with plastic to keep the temperature inside wet for 24 h. They were cured in water after demolding for 28 days in room temperature conditions. Figure 2 shows some photos of the specimens made in this work.

Figure 2. Specimens made.

2.3. Test Procedures

2.3.1. Compressive Strength

Compressive strength tests were carried out at 7 and 28 days in accordance with EN 196-1 and the measurements were carried out using an automated brake and compression resistance tester with a maximum power of 300 kN. The compressive strength was obtained from an average of three tests.

2.3.2. Bond Strength

Each specimen consisted of a mortar cube, 150 mm on each edge, with a single 10 mm diameter steel rod embedded vertically along the central axis for the bond strength test. The steel rods used were hot rolled deformed bars with deformations on their surface in the form of ribs. One-year bond strength was subsequently evaluated by performing a direct pull-out test on a 1000 kN capacity microcomputer control electro-hydraulic suit universal testing machine (WA VW-1000kN) in accordance with the provisions of ASTM C2344 (ASTM 1988). The average bond strength was calculated according to the following formula:

$$\tau_{av} = \frac{F}{(\pi \times d \times l)} \quad (1)$$

where l is the embedment length (mm), F is the force (N), d is the diameter of the reinforcing bar (mm), and τ_{av} is the average bond strength (MPa). The bond strength measurements were performed using three specimens of each mixture.

2.3.3. Drying Shrinkage

Drying shrinkage was measured according to KS F 2424. On three 4 cm × 4 cm × 16 cm mortar specimens with gage stud bolt embedded at both ends of the specimens, measurements of length changes were taken for each mixture. The mortar specimens were cured for 1 day, after demolding, at 20 ± 3 °C in air. Then, changes in length were measured several times (t) and the shrinkage was calculated using Equation (2) at any time up to 60 days:

$$\varepsilon = \frac{\Delta l(t)}{L} \quad (2)$$

where, $\Delta l(t)$ = difference between reading length at time t (days), L = initial reading length at 24 h after demolding (mm) and ε = drying shrinkage (mm/m).

2.3.4. Porosity Accessible to Water

Water-accessible porosity was measured by hydrostatic weighing in accordance with NF EN 18-459. Measurements were made after storage in water for three specimens of 4 cm × 4 cm × 16 cm, for each mixture, at room temperature for up to 28 days. The difference in mass between a sample in the dry state and the same sample in the saturated state was calculated.

For the calculation of the porosity accessible to water P , the following formula was used:

$$P = \frac{M_{air} - M_{dry}}{M_{air} - M_{water}} \quad (3)$$

where, M_{air} = Mass of the soaked sample (g), M_{dry} = mass of the dry sample (g), M_{water} = Mass of the sample immersed in water (g), and P = porosity accessible to water (%).

2.3.5. Rapid Chloride Migration

The 28-day chloride resistance of mortars was assessed by Rapid Chloride Migration (RCM) in compliance with NT Build 492. The specimens for the test were 100 mm × 50 mm disks. First, the disks were saturated with lime-saturated water and then the specimens were subjected for a certain period of time to an electrical potential. After that, by spraying 0.1 M silver nitrate solution, the discs were fractured and the chloride ingress depths were obtained. The chloride diffusion coefficient can be calculated accordingly on the basis of the applied electrical potential, test time, and temperature before and after the test. Each reported value was the average of measurements of three tests for all mixtures.

2.3.6. XRD Analysis

The mineralogical analysis of the materials used, which were reduced to powder, was carried out using the X-ray diffraction method. This method enables the various crystalline phases that make up the material to be defined. Each mineral has a characteristic X-ray diffraction spectrum, depending on the incidence 2θ of the X-ray source on their crystalline planes.

2.3.7. SEM Analysis

Microstructural analyzes were carried out at 14 months, mainly to verify the ITZ of the mortar samples. All the selected pieces were coated with epoxy resin and polished prior to the test. A back-scattered electron (BSE) detector was used to record micrographs. The working distance was set in the range of 10.2–14.8 mm for the SEM test, while the acceleration voltage was 20 kV.

3. Results and Discussion

3.1. Compressive Strength

The compressive strength of the tested specimens is shown in Figure 3. Indicated values are the average of three tests. With increasing rubber content, a progressive decline in compressive strength can be observed. There are two key reasons for the decrease in rubberized mortar strength. Firstly, the low elasticity modulus and the low resistance of the rubber particles mainly decrease mortar strength [43]. Secondly, the particles of rubber are highly hydrophobic, thereby increasing the porosity of the concrete matrix and thus reducing the strength [25,44]. The addition of SF and BFS may increase the rubberized mortar's compressive strength. Replacing 8% of cement by SF increased the compressive strength of the 5RW mix by 8.30% and 18.43% for 7 and 28 days, respectively, while replacing 20% of cement by BFS increased the compressive strength of the same mix by 15.58% and 21.32% for 7 and 28 days. Similarly, substituting 8% of cement by SF increased the compressive strength of the 10RW mix by 28.36% and 12.14% for 7 and 28 days, respectively, while substituting 20% of cement with BFS increased the compressive strength of the same mix by 30% and 28.93% for 7 and 28 days, respectively.

The increase in compressive strength following the addition of SF is due to its filling and pozzolanic effects [33,45]. This observation is attributed to the matrix densification and improvement of the interface of the rubber-cement paste due to the filling effect and silica fume pozzolanic reaction products. The surface treatment of rubber particles with NaOH and silica fume used as cement volume replacement contribute to the improvement of the interface of the rubber-cement matrix [46]. The role of silica fume in improving strength results is primarily due to the interaction of the free lime released by OPC clinker with

reactive silica fume. This leads to the formation in the samples of additional quantities of calcium silicate hydrates, resulting in an increase in the strength values of the RW+SF cured mortars. Compared to the 15% RW mix, the combination of 8% SF and 20% BFS with 15% RW increases the compressive strength by 26.30% and 24.63% for 7 and 28 days, respectively, but compared to the reference mortar, there was a decrease of 63.63% and 59.98% for 7 and 28 days, respectively.

Figure 3. Compressive strength of the tested specimens.

3.2. Bond Strength

Figure 4 shows the pull-out test results of all mixtures after one year of curing. The bond strength for the rubber waste mortar is 18.58 MPa, 16.35 MPa, and 13.88 MPa, for the 5RW, 10RW, and 15RW mixture, respectively, while the bond strength of the reference mortar was 28.84 MPa, a decrease of 35.57%, 43.30%, and 51.87%. The effect of rubber was to decrease the strength of the bond, because when the rubber particles were added into the mixture [47], the effective bond surface area of the specimens was reduced. The weak adherence between the cement paste and rubber particles may be due to this negative effect of rubber on the bond strength. According to [48], the flexibility of the rubber aggregates, the weak interfacial bond between the rubber aggregates and the cement paste, and the interface cracking result in decreased friction between the reinforcement steel bar and its surroundings. According to [49], the reduction of the compressive strength due to the replacement of mineral aggregates with rubber particles has a direct influence on the bonding properties, since the compressive forces in the concrete cover resulting from the wedge action are limited to a fraction of the compressive strength and the rebar-rubberized concrete interface, on the other hand, is often potentially subject to other types of friction than standard concrete, with rubber particles clamping the rebar ribs and providing various mechanisms of energy dissipation.

The 5RW+8SF and 5RW+20BFS combinations show a decrease in bond strength of 65.56% and 50.62%, respectively, compared to the Ref. mixture and a decrease of 46.55% and 23.35%, respectively, compared to the 5RW mixture. The 10RW+8SF and 10RW+20BFS shows a decrease of 56.20% and 43.55%, respectively, compared to the Ref. mix and a decrease of 22.75% and 0.4%, respectively, compared to the 10RW mix. For the 15RW+8SF+20BFS combination, there was a decrease of 56.93% and 10.51%, respectively, compared to the Ref. and 15RW mixtures. It is clearly noted that the addition of additives did not present a positive effect on the steel-cement matrix bonding as regards compres-

sive strength. According to [50], there is a correlation between pull-off test results and compressive strength.

Figure 4. Bond strength of the tested specimens.

3.3. Drying Shrinkage

Figure 5a shows the change in drying shrinkage measured on all mortars as a function of time. This characteristic increases over time due to the progressive loss of capillary water from the mixes. The rubber aggregates incorporated in the study mixes increase the drying shrinkage at all ages with the increase in their contents compared to the mix without rubber aggregates. The increase in drying shrinkage may be due to the higher void content introduced by the incorporation of the rubber aggregates, which results in a porous mortar, as outlined [51–54]. The increase in drying shrinkage is caused by the evaporation of free water from macro pores. The rubber, in particular, may alter moisture diffusion through the matrix as well as the restraining effect of the rubber particles. Because the rubber aggregate exhibits a lower stiffness than the paste (and the sand aggregate), it may increase drying shrinkage. Figure 5b shows the effect of silica fume and blast furnace slag on the evolution of the drying shrinkage with 5% RW. It can be seen that with the addition of SF and BFS, the drying shrinkage decreases with increasing time. The opposite effect was observed beyond 34 days; the decrement of drying shrinkage is more significant in mixtures with addition of SF.

On the other hand, in Figure 5c, with silica fume addition, there is a decrease in the drying shrinkage compared to the 10RW mix, but an increase is observed compared to the reference mixture. The opposite effect is seen with the addition of blast BFS, as a reduction in drying shrinkage compared to both the 10RW mix and the reference mix was promoted by the addition of BFS. It can be noted that, unlike SF, BFS presents a positive effect on drying shrinkage. The effect of the RW, SF, and BFS combination on the evolution of the drying shrinkage is shown in Figure 5d. It can be seen that with the addition of SF and BFS with 15% RW, the drying shrinkage decreases with time compared to the 15RW mixture and the reference mixture. However, the opposite effect is observed after 28 days: with the combination of SF and BFS with RW, the drying shrinkage increases compared to the reference mortar. The shrinkage is sensitive to the evaporation of the free water from the

mixtures and the change of the shrinkage behavior may be related to the change of the pore structure and size distribution of the matrix, which affected the evaporation rate of the mixes.

Figure 5. Drying shrinkage of the tested specimens: (a) mortars containing 5%, 10% and 15%RW, (b) 5% RW with SF and BFS, (c) 10% RW with SF and BFS, (d) 15%RW+8%SF+20%BFS.

3.4. Porosity Accessible to Water

Figure 6 shows the results of the porosity accessible to water of all mixtures. A general increase in porosity is shown by the increase in rubber aggregates content in natural sand volume replacement compared to the reference mortar. Similar results are reported in other studies carried out on RW-based mortars [37] and rubberized concrete [46,55,56]. The order increases are 24.55, 28.22, and 30.42% for 5RW, 10RW, and 15RW, respectively, compared to the reference mortar. This increase can be linked to the non-polar character of the rubber aggregates according to [57,58] and the trapped air trapped during the rubber compound mixing process according to [59]. At the interface between the rubber aggregates and the cement paste, this phenomenon generates porosity.

For mortars based on the combination of SF and BFS with RW, the results of water-accessible porosities indicated reductions of 11.78 and 14.89%, compared to the 5RW mixture, and of 6.19 and 11.39%, compared to the 10 RW mixture, with the addition of 8% SF and 20% BFS, respectively. This effect is mainly due to the increased quantity of hydration products produced by the pozzolanic reaction of SF and BFS with Portlandite, which is deposited within the cement matrix in the open pores and leads to a decrease in total porosity. The effect of the RW+SF+BFS combination on the water-accessible porosity shows a reduction of about 9.5% compared to the 15RW mixture. It is suggested that this decrease can be attributed to the filler effect of the BFS and SF during the mixing of the blends, as well as the formation and subsequent accumulation of hydration products within the available pore spaces of the hardened OPC+SF+BFS mortars, resulting in the pore refinement and a consequent decrease of total porosity.

Figure 6. Porosity accessible to water of all mortars.

3.5. Rapid Chloride Migration

The relationship between the coefficient of chloride diffusion and the mortar composition is shown in Figure 7. It can be seen that the coefficient of diffusion of chloride decreases with the addition of RW (5%, 10%, and 15%) compared to the reference mortar. On the other hand, compared to mortars containing only RW, the diffusion coefficient decreases with the addition of SF and BFS. However, the chloride diffusion coefficient decreases for RW-based mortars with the addition of 5 to 10% RW replacement, further higher replacements up to 15% results in an increased diffusion coefficient. Other researchers have made similar conclusions. Onuaguluchi and Panesar [46] reported the rapid chloride permeability of concrete is reduced by partially replacing crumb rubber (size 86% smaller 2.3 mm) with 5%, 10%, and 15% natural fine aggregate by volume. The inclusion of 5% rubber sand led to a decrease in chloride diffusion, whereas the coefficient of chloride diffusion increased when the replacement ratio increased from 5% to 15% [60]. Gupta et al. [61] found that the coefficients of chloride diffusion of rubber powder concrete (rubber powder replaced sand by 20%) and hybrid rubber concrete (rubber powder replaced sand by 10% and rubber fibres replaced remaining sand by 25%) decreased by 24.5% and 21.81%, respectively, relative to NAC, and rubber powder filling effect could restrict the chloride effect. According to Zhu et al. [62], the chloride ion erosion resistance of CRC is significantly higher than that of NAC, especially when the temperature is below 20 °C. From this research, it can be seen that, as mentioned by other researchers, rubberized mortars develop a better chloride ion penetration resistance due to the filling effect and the impermeable nature of the rubber particles. Owing to the higher activity and specific surface area of SF, the chloride binding capacity of the rubberized mortar with SF is stronger than that with BFS. Therefore, these mortars can be recommended as effective materials for the prevention of reinforcement corrosion in various structures.

Figure 7. Chloride diffusion coefficient of all mortars.

3.6. XRD Analysis

The XRD patterns of OPC, OPC+8%SF, OPC+20%BFS and OPC+8%SF+20%BFS pastes composites at 28-day curing age are shown in Figure 8. In OPC pastes, Portlandite, Calcite, Lime, Calcium silicate-hydrate (C-S-H), and Quartz have been observed. These phases are commonly observed in hydrated cement for 28 days [63]. While $\text{Ca}_54\text{MgAl}_2\text{Si}_{16}\text{O}_{90}$ and Ettringite were detected, lime and calcium silicate-hydrate (C-S-H) were suppressed with SF addition. $\text{K}_2\text{Ag}_4\text{S}_3$ was detected when BFS was added, while Lime was suppressed. The addition of SF and BFS combinations led to the suppression of Lime and the appearance of $\text{Ca}_54\text{MgAl}_2\text{Si}_{16}\text{O}_{90}$. At the same time, with the addition of this combination, it can be observed that the quantity of portlandite has significantly decreased (almost disappeared).

Figure 8. XRD patterns of: OPC, OPC+SF, OPC+BFS and OPC+SF+BFS pastes composites at 28 days.

3.7. SEM Analysis

In order to analyze the influence of RW, SF, and BFS particles on the microstructure of rubberized mortars, BSE tests were performed on five selected mixtures at 420 days. The photos of BSE obtained are presented in Figure 9. Figure 9a–e show the reference mortar, the 10%RW mortar, 10%RW+8%SF, 10%RW+20%BFS, and 15%RW+8%SF+20%BFS, respectively, and images with 100× magnification were taken at several different locations. In the cement matrix, the natural sand is embedded and, as shown in Figure 9a, the interface between the sand and the matrix is compact for reference mortar. Unhydrated/partially hydrated cement particles represent the white (portlandite) and intermediate grey (clinker) elements.

Figure 9. BSE micrograph of mortar samples at magnification of 100: (a) Ref, (b) RW, (c) RW+SF, (d) RW+BFS and (e) RW+SF+BFS.

The darker features are rubber waste. Poor adhesion between the rubber particles and the cement paste was evident (see Figure 9b) (in Mixes with RW). According to [64], this effect could be due either to (a) the absence of bonding and the limited hydration of cement in the ITZ rubber cement paste, (b) the detachment of rubber during the preparation of the specimen, or (c) the combination of the two and (d) the non-polar nature of the rubber surface itself [65]. Interfacial bonding could perhaps be attributed to the compressive and flexural strength reduction [65]. Indeed, the incorporation of rubber aggregates results in a porous mortar likely to be related to the non-polar nature of rubber aggregates in accordance with [57,58] and to the trapped air trapped during the mixing of rubber compounds in accordance with [59]. As illustrated in Figure 9b, this phenomenon generates porosity at the interface between rubber aggregates and cement paste.

Figure 9c,d show SEM observations of 10RW+8SF and 10RW+20BFS samples. The improved integration of the rubber particles in the RW+SF mix (Figure 9c) highlights the filling effect of SF and SEM observations indicate that the use of SF has improved the bonding of the mixtures between the rubber and the cement paste [64]. Rubber particles were better adhered to cement paste, similar to the behavior observed by Pelisser et al. [66] and Gupta et al. [67]. In the cement matrix of the RW+SF and RW+BFS samples, Figure 9c,d show many pores. On the other hand, the images show that the pores are not empty, unlike the samples containing only RW. Such pores are likely to contain SF and BFS, which could explain the improvement of these samples' mechanical and durability features.

Figure 9e shows the same observations as Figure 9c,d, except that for 15RW+SF+BFS samples, the number of pores is more significant but empty. The images also reveal cracks between the cement paste and the fine aggregate/RW across the Interfacial Transition Zone (ITZ) (Figure 9a–e). This cracking can be attributed to the cement paste's shrinkage. Additional support to explain the change in mechanical properties and durability of the rubberized mortar is provided by the above observations.

4. Conclusions

In this study, several important performances of rubberized cement material with additives such as silica fume and blast furnace slag are evaluated and discussed. The results of compressive strength demonstrate that the incorporation of SF and BFS leads to an increase in strength. Substituting 8% of cement with SF increased the compressive strength of the 10RW mix by 28.36% for 7 days and substituting 20% of cement with BFS increased the compressive strength of the of the same mix by 30% and 28.93% for 7 and 28 days, respectively. Compared to the 15% RW mix, the combination of 8% SF and 20% BFS with 15% RW increases the compressive strength by 26.30% and 24.63% for 7 and 28 days, respectively. As shown in SEM images, the use of silica fumes in mortar helps to fill the voids caused by the increase in rubber particles, mainly in ITZ. The introduction of SF and BFS has had a positive effect on the shrinkage of rubberized mortars. Compared to the reference mortar, there is an increase in the porosity of blends made with rubber aggregates. This increase is likely to be linked to the non-polar nature of the rubber aggregates and the trapped air trapped by the rubber compounds during mixing. With the combination of SF and BFS with RW, the results indicate reductions in porosity. The results indicated reductions of 11.78 and 14.89%, compared to the 5RW mixture. This reduction is due to the formation and subsequent accumulation within the available pore spaces of the hardened mortars of hydration products, resulting in the refinement of the total pore system, resulting in a reduction of total porosity. The rapid chloride migration test showed that the resistance of mixtures to chloride penetration was improved by RW. The addition of SF and BFS to mixtures greatly improved resistance to the permeability of chloride. The hybrid rubber mortar is highly resistant to chloride penetration (mixing rubber powder, SF, and BFS).

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