Evaluating damage of rubberized cement-based composites under aggressive environments

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1 # Evaluating damage of rubberized cement-based composites

2 # under aggressive environments

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8 **Abstract:** Rubberized cement-based composites are suitable for large surface applica-

9 tions that require higher strain capacity and lower propensity for cracking owing to length

10 change rather than prioritize strengths. However, bond defects between rubber aggregates

11 and cement matrix affect durability of the composite, especially the sustainability of such

12 materials under aggressive environments. An enhanced rubber-cement matrix bond using

13 a rubber coating solution contributed to a significant improvement in transfer properties.

14 Hence, this study investigated effect of this bond enhancement on resistance of rubberized

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cement-based applications to acid and sulfate attacks. Especially, damage variable was defined as changes of load-resisting areas to evaluate the durability of rubberized mortar specimens during chemical attack process. Results demonstrated that the bond enhancement at rubber-cement matrix interface lowered damage variable of mortar incorporating coated rubber aggregates compared to that of the untreated one. Microstructural analysis still revealed a better interface in coated rubberized mortar exposed to acetic acid and to external sodium sulfate environments.

1 Introduction

Incorporation of rubber aggregates (RA) into cementitious mixtures is detrimental to mechanical properties of rubberized cement-based composites (compressive and flexural strengths, and modulus of elasticity) partly due to bond defects between RA and cement matrix [1–8]. Attempts to improve the bond such as washing or soaking RA with water, pre-treating RA with sodium hydroxide (NaOH), partly oxidizing RA, and coating RA with cementitious materials or sand seem not efficient to improve mechanical properties [1,9]. As emphasized by Pham et al. [10,11], the reduction in strengths can be acceptable owing to low stiffness of RA. Despite strength loss, the composites exhibit high strain

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capacity and low propensity for cracking due to restrained shrinkage [6, 12, 13]. Such behaviors make the materials suitable for large surface applications (pavements and thin bonded overlays) where their strength is not a first priority, but higher deformation before macro-cracking localization is required [12, 14–17]. Nevertheless, due to bond defects at rubber-cement matrix interface, there are limitations on durability of the materials, such as their resistance to aggressive environments. According to Pham et al. [10], solution to coat RA with copolymer before a slight mixing with premixed cementitious mixture induced an enhanced bond at rubber-cement matrix interface, as illustrated in Fig. 1. This bond enhancement results in a significant improvement in transfer properties, such as lower air permeability of coated rubberized mortar [10, 11].

As for the durability of rubberized cement-based composites under aggressive environments, few investigations on resistance of such materials to acid attack have been conducted. Thomas et al. [18] used RA (size 0-4 mm) as a replacement of natural fine aggregates at different levels (0 to 20%). After acid attack, more losses in mass and in compressive strength were observed in control concrete than rubberized ones. For a comparison of the resistance of control and rubberized mortars to hydrochloric acid (HCl) attack, Segre et al. [19] immersed cylindrical specimens (45 mm in height and 30 mm in diameter) in HCl 5% (1.4 M). In order to obtain the rubberized mortars, the authors pre-treated 10% of RA (size 0.2 mm) with saturated NaOH solution for 20 - 30 minutes and then incorporated such treated RA in cementitious mixtures as a partial replacement of cement or sand, by weight. The mass of each mortar specimen was measured after several

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periods of testing time, and the obtained results showed higher resistance of rubberized mortar against HCl compared to the control one in terms of weight loss. On the contrary, Azevedo et al. [20] compared the resistance to attack of a 10% of sulphuric acid solution between control and rubberized concrete specimens (150 mm x 150 mm x 150 mm) and reported that increasing rubber contents resulted in a higher degree of mass loss over a 28-day immersion period.

Similarly, few studies are available on the resistance of rubberized cement-based composites exposed to sulfate attack. Yung et al. [21] investigated anti-sulfate corrosion properties of self-compacting rubberized concrete by comparing weight loss after 5 drying-soaking cycles in sulfate solution. The authors observed that concrete specimens incorporating 5% waste tire rubber powder (size less than 0.6 mm) exhibited sulfate resistance in comparison with the control one. However, large volume of RA incorporation led to nonresistance to sulfate [21]. In order to evaluate impacts of magnesium sulfate (MgSO_4) (3%) on rubberized concrete, Thomas et al. [18] conducted an external sulfate attack test on specimens (100 mm x 100 mm x 100 mm) for 6 months according to ASTM C 1012 standard [22]. At a given time after sulfate immersion, the concrete specimens were weighed, compressed, and checked water absorption. As reported, concrete specimens with higher RA content were more damaged than the control one. As a consequence, more losses in compressive strength and mass, as well as higher water absorption were observed. After 91 days of sulfate immersion, for composites with water-cement ratio of 0.4, compressive strength losses of control and rubberized concretes were 2% and 7%, respectively [18].

From a brief summary above, one can reveal that RA incorporation into cementitious mixtures may be detrimental to the resistance of cement-based composites against aggressive environments. One of typical reasons of low acid/ sulfate resistance may be related to bond defects between RA and cement matrix. In this regard, this paper focuses on effect of rubber-cement matrix bond enhancement induced by rubber coating, as suggested by Pham et al. [10, 11], on anti-acid or sulfate corrosion properties of rubberized mortars. Usually, while some indicators such degraded depth, mass loss, and strength reduction are measured during acid attack, those of the sulfate attack are length gain, mass changes, and strength variations. In this study, damage variable of rubberized mortar specimens under chemical attacks was defined to evaluate effects of rubber incorporation and of copolymer coating. Micro-structural studies using Scanning Electron Microscopy (SEM) were also used to explore any changes at rubber-cement matrix interface in order to explain the durability of rubberized cement-based applications under aggressive environments.

2 Materials and methods

2.1 Materials

RA were incorporated in cementitious mixtures as a replacement of 30% by volume of rounded siliceous sand. Such low stiff aggregates are hydrophobic thus air-entrapment phenomenon happens on the surface of RA when they are in contact with mixing water. The air bubbles are further partly released into the matrix and such phenomenon results in

higher air content of rubberized mixtures. Density and water absorption of sand are 2.62 and 1.9%, respectively. Due to low specific gravity of RA (1.2), viscosity agent made from a high molecular weight synthetic polymer and a polycarboxylate-type superplasticizer were added to prevent segregation phenomenon and to maintain focused workability of fresh rubberized mixtures, respectively.

Table 1 presents the studied mortar mix proportions. It should be noted that acronyms UR and CR stand for mixtures incorporating Untreated Rubber and Coated RA, respectively; letter P refers to coPolymer, which is used as a treatment material to improve rubber-cement matrix interface [10, 11]. For coating procedure, it was followed as recommended by Pham et al. [11]. That is, RA were firstly mixed with styrene-butadiene copolymer (2% of RA by mass). The coated-RA were then maintained under room conditions (20 °C temperature and 50% relative humidity) during 1 hour to condense and stabilize copolymer on rubber surface before a short and light mixing with the pre-mixed cementitious mixture.

Cylindrical specimens used for acid and sulfate attack tests are 28 mm and 60 mm in diameter, as well as 60 mm and 120 mm in height, respectively. All mortar specimens were demoulded 24 hours after casting and then placed in a curing room where temperature and relative humidity were fixed at 20 °C and 95%. At the end of this curing period (28 days), the top and bottom surfaces of cylindrical specimens were ground to impose high parallelism and good contact with platens of the loading machine. They were then coated with a layer of impermeable resin to only obtain the radial acid/ sulfate diffusion inside

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₁₁₆ mortar specimens.

2.2   Experimental programme

2.2.1   Acetic acid attack

Acid attack test was performed according to ASTM C267-01 standard [23]. In NF EN
206-1/CN standard [24], the XA exposure class classifies the environmental conditions
from low (XA1) to high (XA3) risk of chemical attack, depending on the concentration of
aggressive substances from natural soils and groundwater. The most unfavorable exposure,
the class XA3, corresponds to a case where pH of aggressive conditions is ranged between
4.0 and 4.5. Based on a stability of acetic acid in such pH range [25], such acid with pH of
4.0 was chosen. The acetic acid solution (CH_3COOH) having concentration of 0.28 M was
prepared with a solution to specimen volume ratio in the range of 20 to 30. After stirring
distilled water and acetic acid, approximately 2 g/l of NaOH agent was then added to
adjust the pH of the obtained solution up to 4. Cylindrical mortar specimens at the end
of a 28-day curing period were wiped to eliminate superficial water before immersing in
the acid solution. The pH was then checked regularly and maintained at 4 during the
whole experimental period by changing the acid solution every 2 days for the first 10 days,
and every 1 week until the end of this experiment (7 weeks). The process of preparing
mortar specimens and acid immersion is shown in Fig. 2.

2.2.2 External sodium sulfate attack

Sulfate attack test on untreated and coated rubberized mortars was carried out using ASTM C1012/C1012M-18a standard [22]. Cylindrical specimens were placed in a tank and immersed by a sodium sulfate solution, as illustrated schematically in Fig. 3. It should be noted that the solution was prepared by adding 50 g of sodium sulfate (Na_2SO_4) to 1L of distilled water. The pH of such solution was maintained between 6 and 8 with the aid of a pH regulator injecting sulfuric acid (H_2SO_4) (0.05 mol/L). The solution in a tank was kept homogeneous by slightly agitating by a mini-pump. The volume ratio between the solution and specimens was maintained between 3.5 and 4.5 according to the recommendations of ASTM C1012 standard [22]. The sulfate solution was weekly renewed for the first 2.5 months and then for every 2 weeks until the test end. The sulfate concentration was reported to decrease with time of immersion. Therefore, changing solution shortly will ensure higher sulfate concentration in order to accelerate degradation process [26]. It is one of relevant methods to shorten immersion time of sulfate attack.

2.2.3 Compressive test

Load-bearing capacity of mortars at different time of acid/ sulfate immersion was determined according to NF EN 1015-11 standard [27] using a compressive machine with maximum capacity of 100 kN. The loading rates used for compressive tests on acid/ sulfate attacked specimens were 0.2 kN/s and 0.5 kN/s, respectively. Each value of compressive strength was averaged from 3 tests.

2.2.4 Microstructural analyses

Microstructural studies aim to observe any changes at rubber-cement matrix interface under acid/ sulfate attack. For the case of specimen attacked by acid, fractured samples were used. Whereas, polished specimens were prepared for sulfate-attacked mortars. In order to carry out these analyses, it is important to cut perpendicularly the lateral surface of sulfate-attacked specimens to get samples (10 x 10 x 15 mm^3) close to the exposed surface. After air-drying these samples, they were then polished with SiC papers 320, 600, 1200 and 2400 grit for approximately 2 minutes each. The samples were cleaned and then coated by a thin carbon layer right before starting SEM observations.

3 Results and discussion

3.1 Compressive strength

Changes in compressive strengths of acid-attacked mortars are presented in Fig. 4. It can be clearly seen that compressive strengths of mortars decreased with an increase of immersion time, especially a sharp drop of the control mortar compared to rubberized mortars. These findings are in good agreement with conclusions of Thomas et al. [28] who indicated that more loss in compressive strength was observed in control concrete than that of the rubberized concrete. The residual compressive strength of the control mortar was much lower than the one of rubberized mortar, namely 26% after 4 weeks of immersion. The one incorporating coated RA showed a higher residual compressive

₁₇₃ strength (42%) compared to untreated rubberized mortar (32%).

₁₇₄ After a given time of exposing untreated and coated rubberized mortars to sodium

₁₇₅ sulfate solution, the compressive test was conducted. Fig. 5 shows reduction in compres-

₁₇₆ sive strength versus time of sulfate immersion. Such strength loss for the first period was

₁₇₇ more significant than the later time. It seems that the sulfate diffusion becomes lower

₁₇₈ as increasing immersion time due to the fact that products formed close the points of

₁₇₉ entrance.

₁₈₀ ## 3.2   Damage variable

₁₈₁ A scalar damage variable to describe the distress of a material was introduced by Kachanow

₁₈₂ [29]. This scalar can be calculated based on a property that depends on the damage of the

₁₈₃ material, such as stiffness e.g. [16]. It is a relevant parameter to evaluate distresses due

₁₈₄ to internal or external loadings. In this study, as for specimens attacked by acetic acid

₁₈₅ or external sodium sulfate, damage variable (D) is defined and evaluated as a change in

₁₈₆ load-resisting area, as given in Eq. (1). It should be noted that, with regard to chemical

₁₈₇ attack, mortar samples were laterally damaged and most useful test set-up cannot be

₁₈₈ implemented.

$$D = 1 - \frac{\tilde{A}}{A} \tag{1}$$

₁₈₉ where A is initial cross-sectional area (m^2). $\tilde{A}$ is an effective cross-sectional area or an

₁₉₀ equivalent undamaged sectional area of damaged specimen (m^2). The equivalent load-

₁₉₁ resisting area $(\tilde{A})$ consists of an un-attacked part and chemicals-attacked regions close to

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undamaged area that can be still resisted to compressive loading. Herein, one keeps in

mind that damaged areas exhibit a residual strength that should be taken into account in

evaluating $\tilde{A}$. $\tilde{A}$ is obtained based on a statement that strength (f_t) of mortar specimen

before chemical attack is equal to the one of equivalent undamaged parts of chemical

attacked sample, as illustrated in Eq. (2).

$$f_t = \frac{F}{A} = \frac{F^*}{\tilde{A}} \tag{2}$$

From Eq. (2), $\tilde{A}$ is determined as Eq. (3), where F and F^* are the maximum compressive

bearing capacities of a sound specimen and the one having undergone the chemical attack,

respectively (kN). F and F^* are determined experimentally. All parameters are described

in Fig. 6.

$$\tilde{A} = A.\frac{F^*}{F} \tag{3}$$

The evolution of damage variable (D) during acid attack for control mortar and rubber-

ized mortars is represented in Fig. 7. Damage variable increased quickly and differentially

between the control mortar and rubberized mortars. Initially, damage variable of the

control mortar seemed to be linear-developed during the first four weeks of immersion.

A slight rise of damage variable after its sharp increase for a given time of immersion

has demonstrated that acid-attacked area of rubberized mortars can be partly of loading

resistance. Damage variable values of coated rubberized mortar (30CR-P) illustrated in

Fig. 7 is always lower than that of the untreated one (30UR). Lower damage variable of

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30CR-P is attributed to the rubber-cement matrix bond enhancement.

The difference in damage variable (D) under external sodium sulfate attack between untreated rubberized mortar and the one incorporating copolymer-coated RA is presented in Fig. 8. Damage variable was observed to increase with increasing time of sulfate immersion. Moreover, untreated rubberized mortar always exhibited higher damage variable than the coated one and it can be explained by an improved bond at rubber-cement matrix interface that reduces the ingress of damaging agents.

3.3 Changes at rubber-cement matrix interface

In degraded areas induced by acid attack, it can be clearly seen that the interface between untreated RA and cement matrix seems to be still porous as it is generally observed in untreated rubberized mortar (Fig. 9a). In case of the one incorporating coated RA, the degraded cement matrix still bonds to the coated RA (Fig. 9b). Such characteristic can result in a decrease of acid diffusion into coated rubberized mortar, leading to reduction in its strength loss.

A general observation on microstructure of rubberized mortars close to the sulfate-exposed surface shows micro-cracks in both untreated and coated rubberized mortar specimens (Fig. 10). It is a consequence of tensile stress induced by formation of expansive products such as gypsum or ettringite. As observed in Fig. 10a, the gap around untreated RA appeared to be filled by such crystals more than that of the coated one. It could be attributed to bond defects at untreated rubber-cement matrix interface, leading

to high sulfate diffusion into the composite via porosity at the interface. For the case where coated RA were used, no clear difference in sulfur distribution within the interface and other neighboring areas was noticed (Fig. 10b). The enhanced interface and effect of copolymer coating may be responsible for these findings. Interestingly, despite a good interface between sand and cement matrix, expansive products were still generated and the density of sulfur at such interface appeared to be higher (Fig. 10c) than that at coated rubber-cement matrix transition zone (Fig. 10b).

Due to low stiffness of RA, their presence in composites can partly delay cracking phenomena due to expansive crystals at rubber-cement matrix interface more than natural aggregates. Hence, cracks can be more visible at sand-matrix interface than that encountered with RA, especially with the ones coated by copolymer (Fig. 11).

4 Conclusions

From experiments focusing on the effect of an enhanced rubber-cement matrix interface on the durability of rubberized mortars under aggressive environments, the primary conclusions can be summarized as following:

- Rubberized mortars behave better than the control one in preventing distresses due to acid diffusion into the composite, especially when rubber-cement interface was enhanced by copolymer coating. Such improvement in the durability can be attributed to reduced water capillary absorption of rubberized mortars and especially to lower

permeability of the coated one.

- Under external sodium sulfate attack, higher reduction in compressive strength was noticed in untreated rubberized mortar compared to the one incorporating copolymer-coated RA. After an exposure to external sodium sulfate solution, the rubber-cement matrix interface seems still sound.

- To evaluate the resistance of mortar to aggressive environments, damage variable is defined as relative change in equivalent load-resisting area. Based on damage variable values, coated rubberized mortar appeared to be more durable than the one incorporating untreated-RA against chemical attacks.

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Figure 1: Effect of copolymer coating on rubber-cement matrix interface: (a) bond defects in untreated rubberized mortar (UR-Untreated RA), and (b) bond enhancement between coated RA (CR-P) and cement matrix (C) [11]

Figure 2: Moulds and specimens (a), grinding specimens (b), coating resin on top and bottom surface of specimens (c), immersing specimens in acid solution (d)
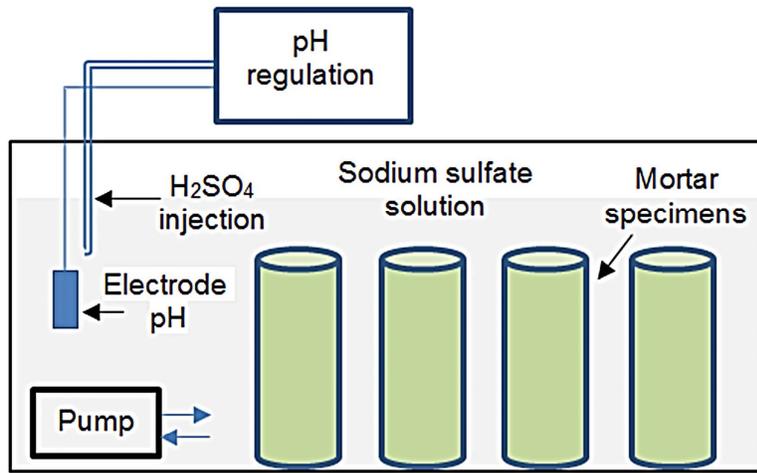
Figure 3: Experimental set-up of external sodium sulfate attack test
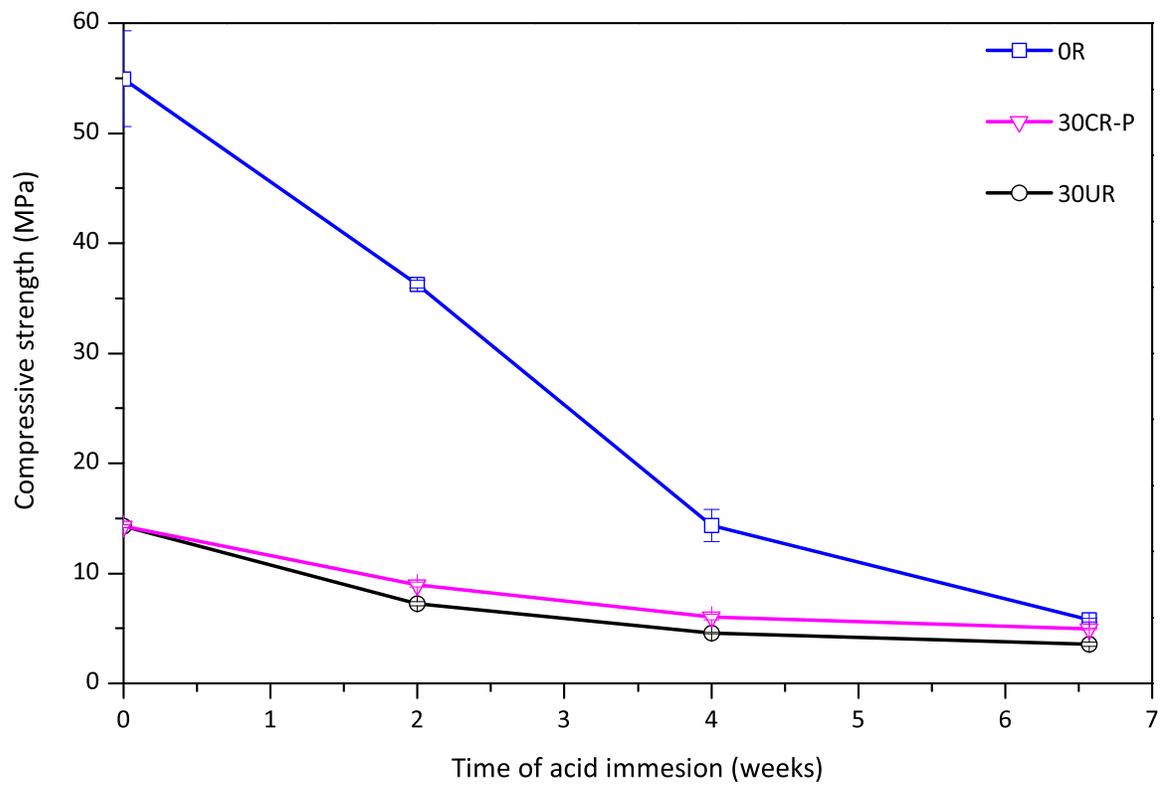
Figure 4: Changes in compressive strength versus time of acid immersion
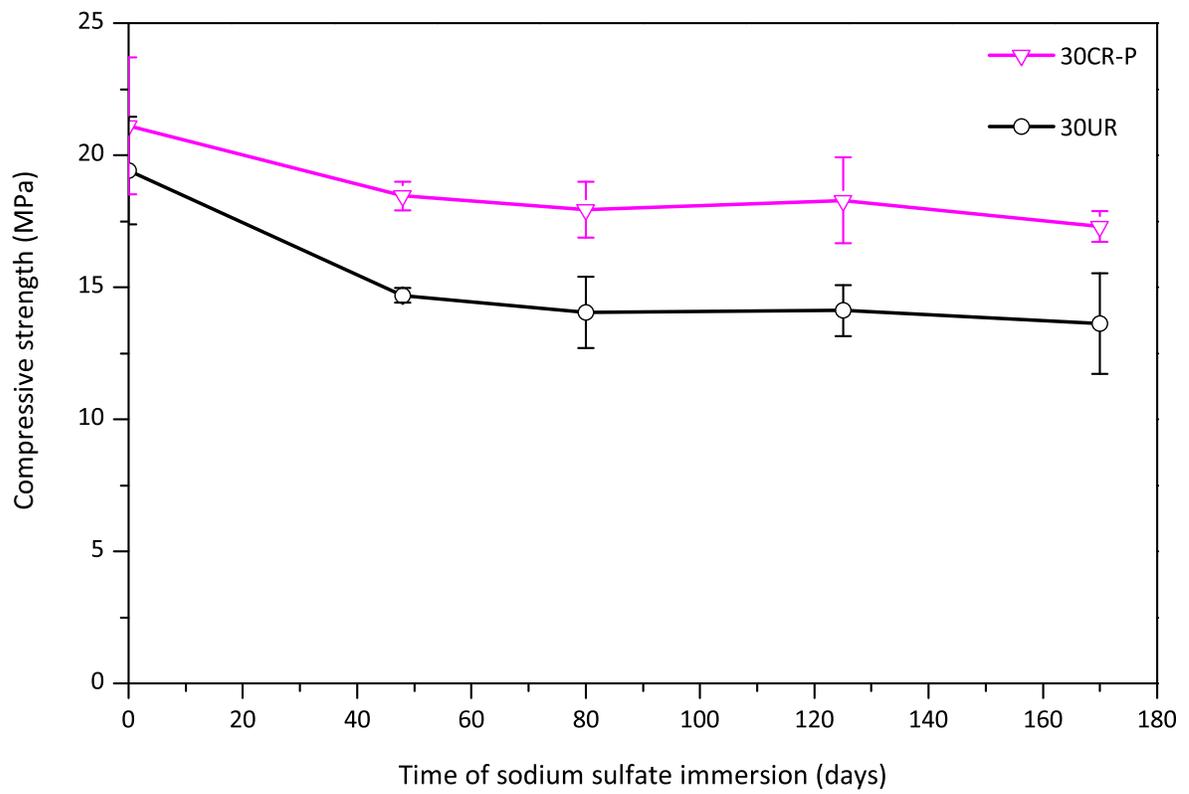
Figure 5: Changes in compressive strength versus time of sulfate immersion
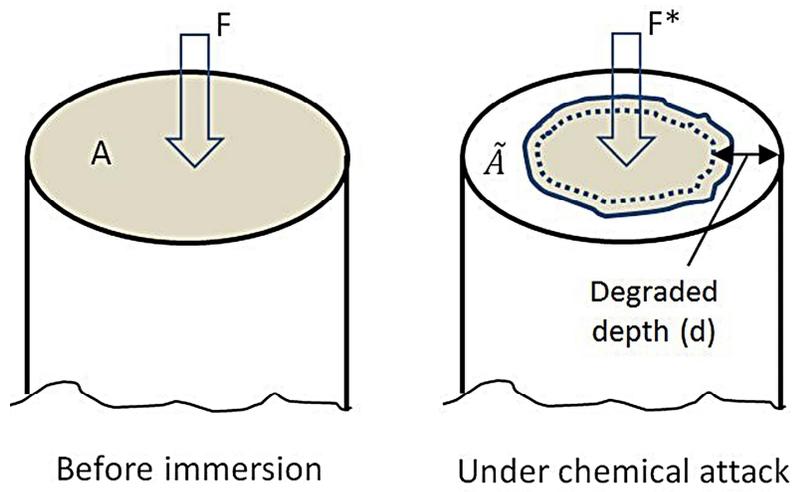
Figure 6: Illustration of determination of equivalent load-resisting area ($\tilde{A}$)
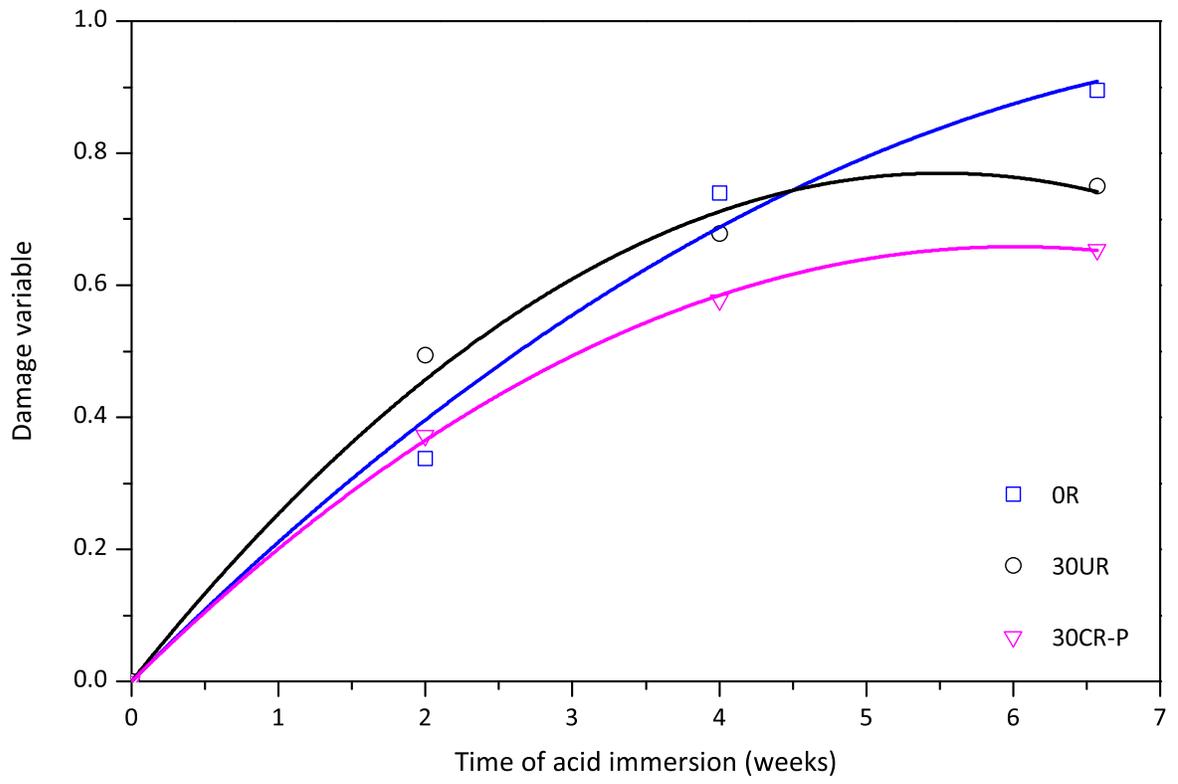
Figure 7: Damage variable due to acetic acid attack
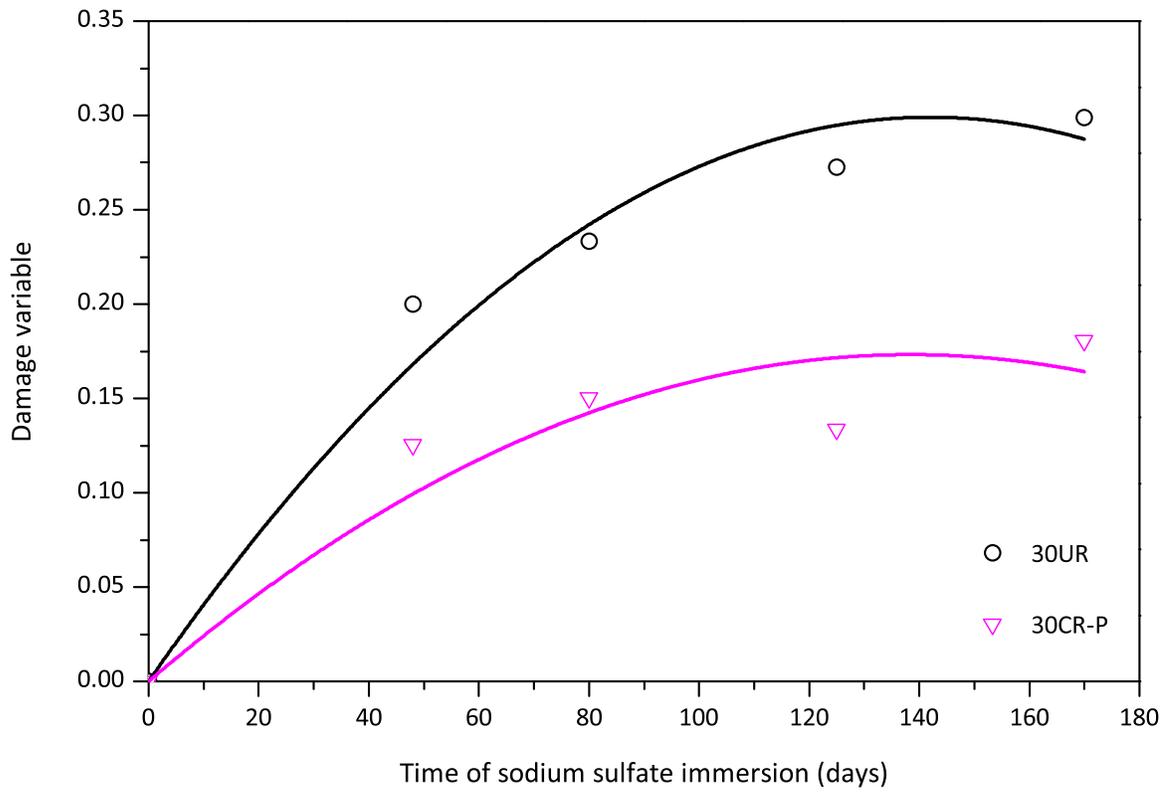
Figure 8: Damage variable due to external sodium sulfate attack
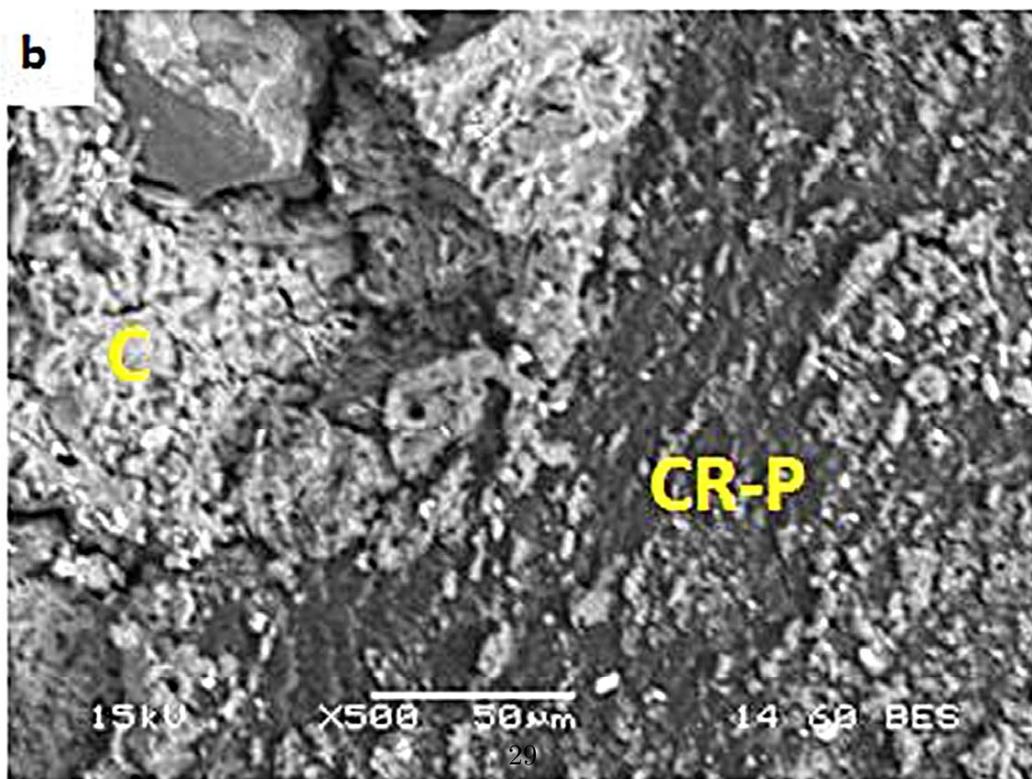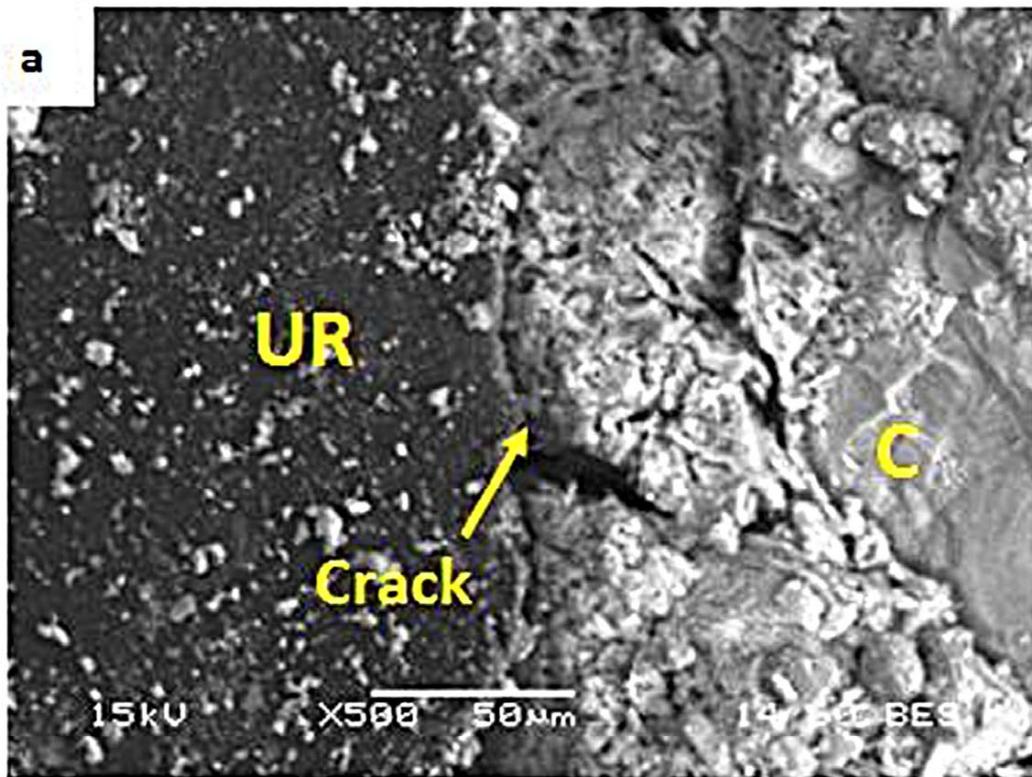
Figure 9: Changes at rubber-cement matrix interface in degraded areas induced by acid attack: (a) Untreated rubberized mortar, and (b) Coated one
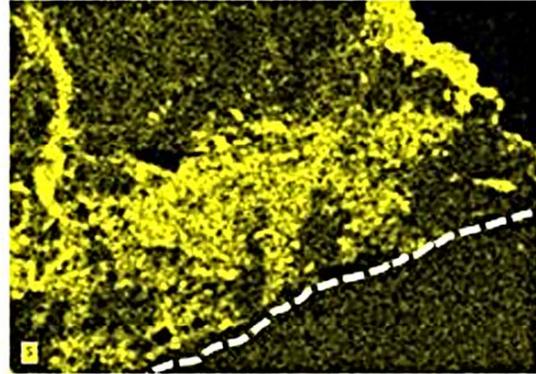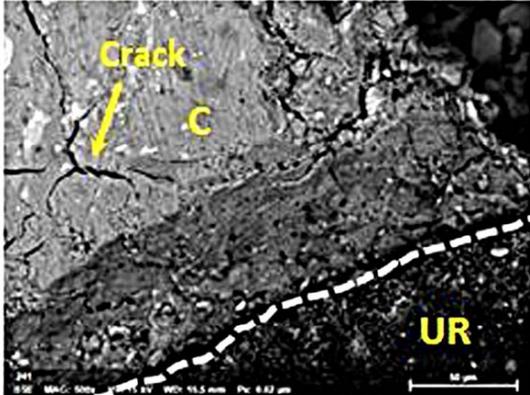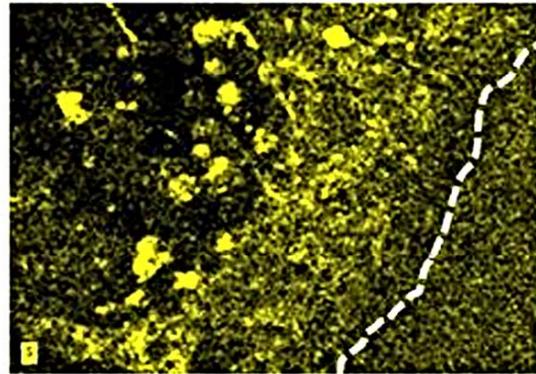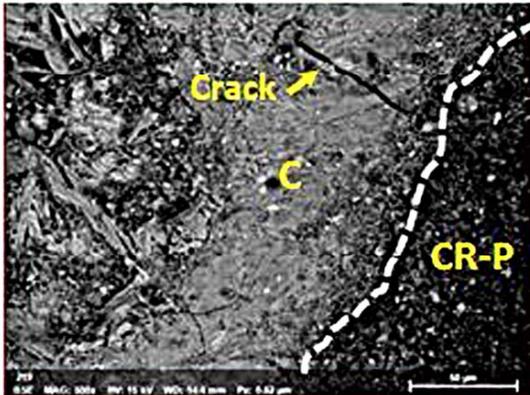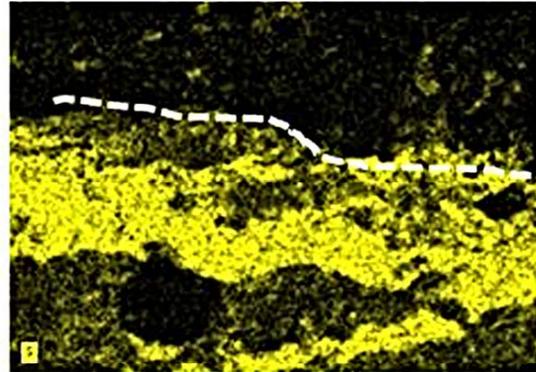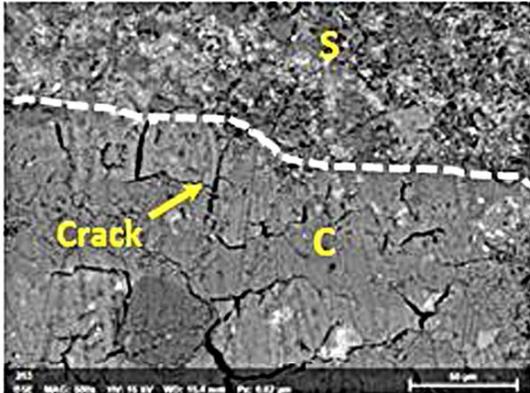
Figure 10: Sulfur distribution at the interfaces between cement matrix and: (a) untreated RA, (b) coated RA, (c) sand (BSE x 500)
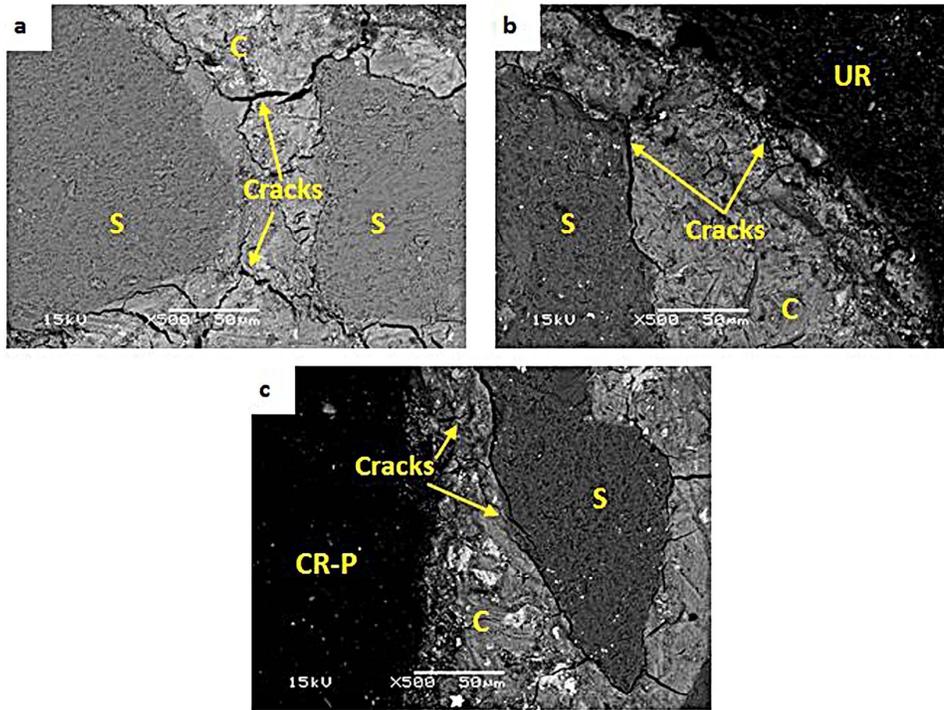
Figure 11: SEM images (x500) indicating micro-cracks induced at (a) sand-cement matrix interface, in (b) untreated rubberized mortar and (c) coated rubberized mortar

Table 1: Mix design and proportioning (values in kg/m^3)

Mix name	Cement (CEM I 52.5R)	Sand (0-4mm)	Water	RA (0-4mm)	Superplasticiser	Viscosity agent
0R	500	1600	235	-	3.25	0.9
30UR	500	1120	235	220	3.25	0.9
30CR-P	500	1120	235	220	3.25	0.9