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Review

Current Knowledge and Pending Research on Sulfate Resistance of Recycled Aggregate Concrete

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Abstract: The building sector's sustainability requires construction and demolition waste (CDW) to contribute to the circular economy. Among the CDW, recycled concrete aggregates (RA) have been mainly studied to replace natural aggregates. Still, the approval of their use in regulations and standards is slower. Some barriers to the adoption of RA are related to the durability of recycled aggregate concrete (RAC). However, their physical and mechanical properties have been extensively studied. The durability risks associated with sulfate attacks have been solved for conventional concrete. However, sulfate attack on recycled concrete still raises numerous unsolved questions. In this literature review, the experience of sulfate attack on RAC is compiled and analyzed using a compressive framework highlighting the most relevant aspects of the new matrix in RAC and the old matrix of RA to support its relevance to the damaging sulfate process. Suggestions for further research are presented to understand the full extent of this issue and contribute to incorporating and extending recycled aggregates into existing regulations.

Keywords: recycled concrete; recycled concrete aggregates; sulfate attack; review

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1. Introduction

The construction industry significantly impacts the environment due to its high material and energy consumption. Concrete has a significant environmental footprint because of the raw materials, energy, and CO_2 emissions associated with its production (about 5–7% of global emissions) [1,2]. However, one strategy that can improve concrete's sustainability is the use of CDW as aggregates in its production [2–4]. This approach offers several environmental benefits, including reducing the exploitation of natural resources, decreasing industry and municipal wastes for disposal, and significantly decreasing transportation costs [2,5,6]. Specifically, aggregates derived from concrete demolition, known as recycled concrete aggregates (RA), have been found to possess competitive characteristics for use in the construction industry [7,8].

Today, some building codes allow certain replacement percentages of recycled fine and coarse aggregates in structural and nonstructural concrete production [9,10]. In general, they set limits for the replacement of NA by RA according to the purpose of the concrete (strength class, environmental exposure class) and the source/type of RA (classification) that determines their properties [7,9–11]. Increasing the allowable replacement levels and the wide use of RA in different mixes will largely depend on a thorough understanding of RA's effects on the performance of the new concrete [12,13].

The mechanical and physical properties of RAC and (and RAM) have been extensively studied. Generally, mechanical strength and stiffness are lower for RAC than for NAC due to its higher porosity [14–17]. RAC's transport properties are higher also due to the higher porosity [18–20]. Regarding their durability, although many studies have been carried out [21–25], the wide variety of attacks and methods used to evaluate them requires an exhaustive analysis of experimental campaigns for a complete understanding.

Sulfate attack (in all forms) is a well-known problem in concrete technology [26–32]. Proven technological solutions already exist for producing conventional sulfate-resistant concretes [33–35]. However, RACs (and RAMs) have very different properties from NACs, mainly higher porosity [14,18]. In addition, RAC presents a larger ITZ, which includes the interface between the old cement paste and the new cement matrix [36]. This issue requires a focused study of the sulfate problems for this new material.

For clarity, this paper is organized as follows: Section 2 presents the current state of knowledge on two issues related to the article's subject—sulfate attack and related durability problems and the properties of recycled aggregates and their effects on cement-based materials. Section 3 presents a comprehensive analysis of experimental results, classified by type of sulfate attack (ESA, PSA, and ISA) and, in the case of ESA, by material scale (RAC and RAM). Section 4 discusses the previous two chapters, focusing on the effect of RA, technological solutions to improve the quality of RAC, and recommended guidelines for further research. Finally, Section 5 presents the conclusions of this work.

2. Current Knowledge about Sulfate Attack: The Particular Case of Recycled Aggregate Concrete (RAC)

In the literature, many papers on RAC's durability focus on investigating the transport properties, as they are closely related to most durability issues [14,37–44]. In addition, several papers examine the performance of RAC under specific exposure conditions. Papers on sulfate attacks remain low [45–94], especially considering the large number of parameters that influence attack development. This section aims to provide an overview of the current knowledge of sulfate-related durability problems and highlight the key factors to consider when evaluating this attack on cement-based materials with RA.

2.1. Sulfate-Related Attacks

Sulfate attack is a chemical attack with various forms, usually associated with other types of attack; it is extensively described by the literature and has several technological solutions for conventional concrete [26,32,33,95,96]. However, research on the mechanisms associated with the attack is still ongoing due to its complexity, the number of involved parameters, and the variety of measurement methods [97–99].

2.1.1. Nomenclature

For a long time, the term "sulfate attack" was used to refer to durability problems caused by the sulfate ions in concrete. However, it is possible to distinguish different processes, and the nomenclature proposed by Neville [97] is adopted in this review. External sulfate attack (ESA) is the chemical attack that occurs on hydrated Portland cement paste in the presence of external sulfate ions that ingress into concrete by a transport mechanism. When the concrete components provide the sulfate ions, it is called an internal sulfate attack (ISA). Finally, real field structures exposed to a rich sulfate environment (soil or water) are subjected to moisture gradients, causing physical attack by salt crystallization. When sulfate salt is involved, it can be called a physical sulfate attack (PSA). In the literature, "sulfate attack" usually refers to ESA and PSA, as both phenomena are considered to be durability issues related to an aggressive environment. In contrast, ISA is related to material impurities, mixture design, or manufacturing process issues. The phenomenon known as delayed ettringite formation (DEF) is merely a specific case of ISA.

2.1.2. Mechanisms

External sulfate attack (ESA) is a chemical reaction between some phases of hydrated Portland cement and sulfate ions from the environment. Sulfate ions can react with calcium hydroxide to form gypsum (Equation (1)), while the aluminate phases of cement combine with gypsum and additional sulfate ions to form ettringite (Equation (2)). These reaction products, particularly ettringite, occupy more space than the constituents from which they are derived and are responsible for the physical effects observed in the material [32]. These effects include swelling, cracking, and spalling due to the internal tensile stresses of ettringite precipitation. Swelling occurs due to the crystallization pressure of ettringite formation in a sulfate-supersaturated pore solution, not when all the pore volume of the concrete is filled with ESA products [100]. In later stages, portlandite consumption for gypsum formation leads to instability in the C-S-H, resulting in the softening of the material [26].

$$Ca^{2+} + SO_4^{2-} + 2H_2O \rightarrow CaSO_4{\cdot}2H_2O \quad (1)$$

$$6Ca^{2+} + 3SO_4^{2-} + 2Al(OH)_2^{-} + 8OH + 26H_2O \rightarrow 3CaO{\cdot}Al_2O_3{\cdot}3CaSO_4{\cdot}32H_2O \quad (2)$$

When the cementitious matrix has a significant carbonate content (e.g., due to the use of limestone filler), a further stage of attack develops in which the precipitating mineral is thaumasite. In general, thaumasite formation has been observed under low-temperature conditions, and the damage associated with this condition is more significant than that at ambient temperatures [101,102]. In magnesium sulfate attack, Mg^{2+} competes with ${SO_4}^{2-}$ to combine with portlandite, resulting in brucite precipitation on the concrete surface. This poorly soluble and dense compound acts as a barrier, controlling the ingress of sulfate ions into the material. However, Mg^{2+} ions can also react with C-S-H, replacing the calcium ion and forming M-S-H, a nonbinding gel that produces the softening [103,104].

For moisture gradients in the material, the conditions for PSA are present. This physical attack is merely a particular case of salt crystallization. The damage mechanism consists of the precipitation of sulfate salts in the internal pores of the concrete after these salts have entered from the outside by different transport mechanisms (convection by capillary absorption or pressure gradient, diffusion). The crystallization pressure associated with the precipitation of these salts in the pores of the concrete causes internal cracking of the concrete by tensile stresses, increasing the transport of the aggressive solution in the material for further advancement of the attack [30,105,106].

Finally, the chemical mechanism of ISA is similar to that of ESA, but in this case, the sulfates do not come from the environment but from the concrete components. For ISA damage, internal sulfates and sufficient moisture content must trigger the damaging reactions [107].

Two key factors that determine the performance of concrete against sulfate attack are its transport properties (kinetic condition) and mineralogical composition (thermodynamic condition). A more compact and dense material with lower transport properties is more resistant to penetration by aggressive ions, regardless of the mode of ion transport [108]. In the case of internal sulfate attack (ISA), where ions are already present in the material, a dense concrete would also delay the ingress of additional water, which is necessary to initiate the mechanism [109]. Moreover, the mineralogical composition of the concrete, specifically the aluminate phases and portlandite content in its cement paste, may affect its susceptibility to sulfate attack because these phases can react with sulfate to form harmful products [26,27,110]. In the case of ISA, the sulfate content of the concrete components, including cement and aggregates, is a crucial factor in the development of the attack.

2.1.3. Evaluation Methods

Sulfate performance is usually evaluated by exposing specimens to different conditions and monitoring some characteristic properties over time. Several properties have been used to assess sulfate attack. The most common ones are expansion, mass loss, and drops in mechanical properties (strength and stiffness) [95,111–116], each having advantages and disadvantages but good statistical results. Additionally, instrumental techniques such as XRD, XRF, thermogravimetry, FTIR, and SEM/EDS make it possible to characterize and quantify the profile of attack progress in terms of elemental oxides (i.e., SO_3 concentration) and mineralogical compounds (i.e., phase changes) at different scales (mesostructural and microstructural). There are even works that use electrochemical techniques [49,90], changes in microhardness at the interface [117], or fracture energy [55] to evaluate the progress of sulfate attack.

There is a large amount of variability in mixture performance depending on the test or exposure conditions. Effects such as the temperature, the specimen/solution volume ratio, the shape and size of the specimen, and the cation associated with sulfate, among others, affect the failure mechanisms [96,115]. Two fundamental setup variables are the sulfate concentration and the humidity regimen. The former defines the rate of diffusive transport (concentration gradient), and the latter activates or deactivates the convective transport of the solution (by capillary absorption or a pressure gradient). When specimens are saturated (i.e., immersed in sulfate solution), only ESA occurs [98,115,118,119]. In contrast, when specimens are subjected to wetting and drying cycles or partial saturation, the conditions for combined ESA and PSA occur [106,117,120,121]. The exposure conditions used in the laboratory are generally more severe than those found in the field, as the aim is to accelerate the attack to obtain information within the timescales of an investigation.

2.1.4. Technological Solutions

Technological solutions for sulfate-resistant concrete require physical and chemical improvements to the cement matrix and the ITZ. Physically, this is achieved by reducing the connected capillary porosity of the cement paste; chemically, improvements are made by decreasing the content of reactive phases in the cement. These solutions can be achieved by reducing the w/c ratio (use of chemical admixtures), using cement with low C_3A content, and using active SCM (reduction in clinker factor, pozzolanic reaction). Current regulations have focused on these two solutions to develop sulfate-resistant concretes.

2.2. Characteristics of Recycled Concrete Affecting Performance against Sulfate Attack

2.2.1. RAC Aspects in Relation to Sulfate Attack

Two fundamental aspects of RAC are linked to sulfate attack on RAC: on the one hand, its higher porosity and higher interface content (kinetic aspect), and on the other hand, the high cement paste volume or high sulfate-reactive phases (thermodynamic aspect).

Generally, RAC is more porous than NAC due to the higher porosity of RA [18,22,42,122–126], the higher number of interfaces generated by RA [36,127–130], and the higher amount of entrapped air due to the shape of particles of RA [86,129]. For this reason, the strength and modulus of elasticity of a mixture with RA are often lower than those of the same mixture with NA. Numerous papers have reported higher transport properties of RAC than NAC [14,22,37,42,44,131], which is attributable to its high porosity.

Increased porosity would harm sulfate attack, allowing the ingress of more aggressive ions in concrete subjected to ESA or ESA + AFS or favoring the ingress of the water required for ISA development. However, increased porosity could also have some positive effects. Some recycled concretes performed better under specific proportions and exposure conditions than those with natural aggregates [58,59,62,65,67,81,91,94], suggesting that other positive effects of RA may compensate for the expected adverse effects.

Regarding chemical–mineral properties, the main difference between RAC and NAC regarding sulfate attack is the higher relative cement-paste content due to mortar or cement paste in the RA. Cement paste could contain sulfate-reactive phases (CH, AFm), leading to a more extensive attack. However, the content of additional reactive material would be limited by the ratio of replacement with RA and the attached mortar or cement-paste content of the RA. As an example, considering concrete containing 40% coarse aggregate, 40% fine aggregate, and 20% cement paste by weight, if 100% of the coarse aggregate is replaced by coarse RA containing 20% cement paste by weight (the expected value [132]), the relative cement-paste content in the new concrete would increase to 28%. If, in addition, 100% of the fine aggregate is replaced by fine RA containing 30% cement paste (expected value [133]), the relative cement-paste content in the new concrete would increase to 40%. The potential reactivity of RA with external sulfate ions has yet to be thoroughly investigated and is one of the gaps in the current literature.

2.2.2. RA Properties

The performance of RAC would be affected not only by the replacement ratio of RA but also by their physical and chemical properties. The relevant physical properties of RA are mortar/paste content, porosity, and absorption. These properties are related to the source concrete's properties and the crushing method [23,40,133]. The chemical properties of RA depend on the concrete source, especially the binder composition (cement and supplementary cementitious materials) and its use (sulfate contamination). In addition, the degree of carbonation of the cement paste in RA can also affect the performance of the aggregates when exposed to sulfate environments [22,123]. The content of attached mortar or cement paste has been suggested in the literature as a quality control parameter [134,135]. However, there is no reliable and unified method for this, and no methods have been proposed in the literature to assess the potential reactivity of the phases included within the RA.

Regarding ISA, the key factor for RA is their likely contamination with compounds necessary for the damaging reaction. In particular, the contamination of aggregates with sulfate-based constituents is a recurring problem since, depending on the extraction and separation process quality during the demolition phases, RA may have a high content of gypsum, a material widely used in construction. This problem, addressed by several papers in the literature [45,46,48,51,73,81], raises the question of the permissible limits for sulfate content in aggregates, which are usually limited by existing regulations.

Finally, the moisture content of RA is also an important aspect considered in several studies [15,23,40,136,137]. The main concern is about the higher porosity of RA with respect to NA and its consequent higher water absorption capacity. Depending on the moisture content of the aggregates at the time of mixing, they can change the effective w/c ratio of the mixture, especially at the interfaces, and thus significantly affect the transport properties of RAC. In addition, they can modify the curing process using the internal curing of the concrete [15,48,138,139].

Given the above, the adoption of criteria for the characterization and qualification of recycled aggregates appears to be a possible need, not only for their effect on the general properties of new concrete but also for their effect in the specific case of sulfate attack.

2.2.3. Regulations

Several countries have already included the use of recycled aggregates in their regulations. Most of them establish a quality classification of recycled aggregates according to their composition or parameters, such as absorption and density, and then define the limits of the permissible contents and the purposes for which they can be used (strength level, structural use, type of exposure) [9,11,140]. The regulations generally allow the use of RA in nonstructural or structural concretes exposed to conditions of low aggressiveness to their durability.

Regulations also set requirements for concrete aggregates in general. Among these requirements, most regulations limit the content of contaminants that may affect the durability of concrete, for example, by setting the maximum content of soluble sulfates to values between 0.2% and 1.0% [9,83]. These limits can be restrictive in the case of RA, which are highly susceptible to contamination, depending on the process used to produce the RA or the environment to which the original concrete was exposed. Modifying the regulations to increase the allowable content of RACs increases the possible uses of RACs and the limited SO_3 content, which will depend mainly on the research progress on this issue.

3. Experimental Results Available in the Literature

As mentioned before, the number of research papers on the performance of recycled concrete against sulfate attack still needs to be increased in order to understand the degree of influence of each intervening variable. Although there are several publications on this topic, they vary significantly regarding research objectives, setup parameters, and evaluation methods. Many of these studies focus on characterizing specific types of recycled concrete aggregates (RA) or evaluating eco-efficient concretes that incorporate RA together with SCMs. These studies examine various properties, including sulfate resistance, but cover only a few variables. Some research also considers sulfate attack in combination with other forms of deterioration, such as freezing and thawing. Each paper uses different test conditions and evaluation methods, making comparing results and drawing meaningful conclusions across different studies challenging.

The literature data was first classified based on the predominant type of sulfate attack: ESA, PSA, or ISA. In order to compare the results of studies with different experimental setups and evaluation methods, a numerical factor was defined: the degradation ratio, which is the relation between the degradation observed for each evaluated RAC (or RAM) and its corresponding reference concrete (or mortar) in a given time. One example is the final expansion of RAC divided by the final expansion of the reference NAC (same criteria for compressive strength loss, weight variation, SO_3 incoming, et cetera). Degradation ratios greater than 1 indicate worse performance of RAC compared to NAC, while ratios below 1 indicate better performance.

The variety of existing test parameters explains the large variability in results in the literature. For example, no trend was found when correlating the deterioration ratio with the w/c ratio of concrete. This is because there are statistically very few results, and they come from tests with very different setups (exposure, evaluation method), making the results not directly comparable. This paper presents the degradation ratio in relation to the RA content, which is a commonly used parameter in literature reviews [21–25] and is significant for regulatory discussions. Regarding the ISA, only a few papers are available, and the experimental setups are quite diverse, making them difficult to compare. The key experimental setups and outputs are summarized and described in the text to conduct a qualitative analysis.

3.1. ESA

3.1.1. Recycled Aggregate Concrete (RAC)

Few studies [52,54,56,61,66,67,70–72,75,77,79,89,91] evaluate the performance of RAC against ESA exposure using immersed specimens. In addition, several works [55,56,61,74,77,78] investigate its performance against ESA in conjunction with other durability tests, thus limiting the campaigns and the number of parameters each test covers. Figure 1 shows the processed results of studies that have examined ESA in concrete with CRA.

In general, an increase in CRA content is correlated with an increase in the deterioration ratio. However, the few existing results of partial substitutions do not show a conclusive relationship between CRA content and performance [52,67,75,79]. For 100% CRA replacement, deterioration ratios ranged from 0.62 to 2.25. All cases with a degradation ratio lower than 1 were cases where 100% RA was combined with some SCMs [66,71,72,91]. Then, depending on other parameters such as matrix quality, the degradation ratio of concretes with 100% RA concretes varied between 1.06 and 1.73 (except for Bulatovic et al. [93], with a degradation ratio of 2.25).

The higher porosity of RA explains the increase in RAC deterioration compared to NAC deterioration. In this type of attack, the ingress of sulfate ions into the specimens is due to diffusion, and the rate of diffusion of sulfate ions increases as the total porosity increases [32]. No consistent relationship between RA content and deterioration rate is observed for concretes with partial replacement. Hence, Arafa et al. [79] and Santillán et al. [67] observed a worse sulfate performance for partial RAC replacements (60 and 75%, respectively). Xie et al. [75] report that 30% RAC outperformed the reference, and the ratio increase for values above 50% replacement.

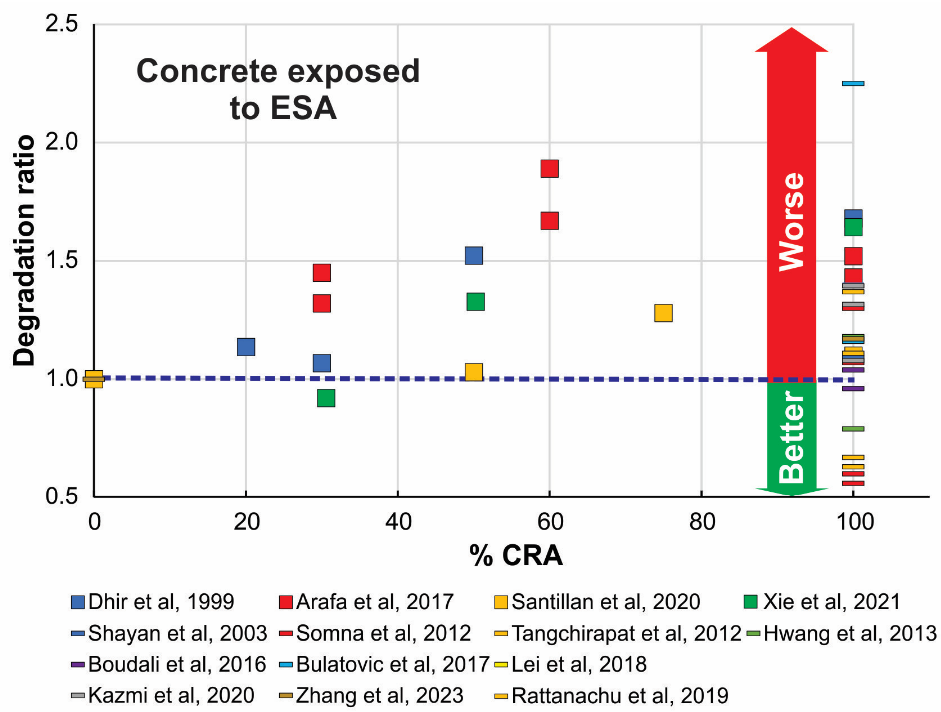

Figure 1. Degradation ratios of concretes with different % CRA exposed to ESA [52,54,56,61,66,67,70–72,75,79,85,91,93].

The results of each experimental campaign show a substantial effect on the quality of the RAC cement matrix. For example, Bulatovic et al. [93] evaluated RAC with different cement types and w/c ratios (good- and poor-quality matrices). The RAC degradation ratio (100% RA) with a poor-quality matrix was 2.25 and 1.16 for sodium and magnesium sulfate exposure, respectively. In contrast, the RAC with good-quality matrices performed similarly to the reference concretes. According to Dhir et al. [52], concretes with a low strength level (w/c between 0.81 and 0.76) showed comparable performance to the reference mixtures for CRA replacement values up to 30%, while for higher replacement values, the deterioration increased, reaching a degradation ratio of 1.68 for 100% CRA replacement.

Some of the quoted papers specifically study the effect of SCM on the performance of recycled concretes. This is the case for Somna et al. [71], Tangchirapat et al. [72], Boudali et al. [91], and Rattanachu et al. [66]. In all of those studies, RACs were observed to have slightly worse performance than NACs (degradation ratio between 1.15 and 1.4), but performance was even better than that of the control when RAC had pozzolanic SCM (degradation ratio < 0.7). The positive effect of pozzolanic SCMs on the performance of concrete against sulfates is well known [35,49,111,141]. The secondary reaction of SCMs consumes the portlandite required for ESA. In addition, it refines the pores by forming C-S-H, significantly reducing the concrete's transport properties. In the case of RAC, equal or even higher efficiency is observed for SCM compared to conventional concrete [65,71,122]. This can be attributed to the possibility of the SCM reacting with the portlandite of the RA in the interface zone, which is the most porous zone of the system.

Results suggest that the use of RA does not have a unique effect on ESA. RAs have some positive effects that can compensate for the negative effect of higher transport properties, and this effect depends on other test parameters. On the other hand, the results indicate robust control of the quality of the matrix in the development of the ESA. Concretes using SCM as a cement substitute had improved performance.

3.1.2. Recycled Aggregate Mortar (RAM)

Figure 2 shows the degradation ratio for the case of FRA mortars exposed to pure ESA. In this case, there are very few results in the literature, and the observed spread is wide.

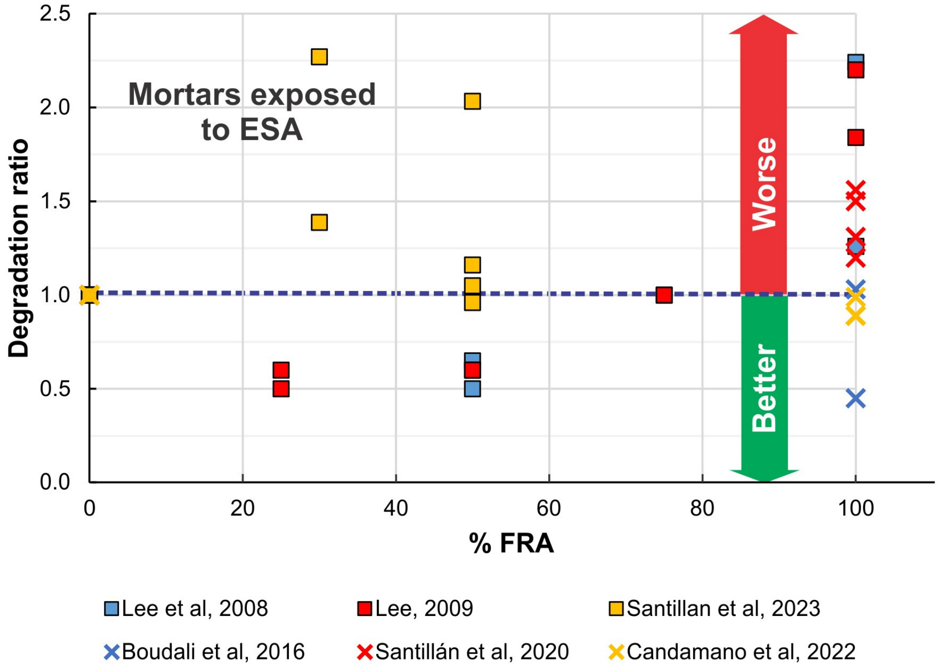

Figure 2. Degradation ratios of mortars with different percentages of FRA exposed to pure ESA [59,60,69,87,91,94].

Lee [59] and Lee et al. [60] report a positive effect of replacing up to 50% of the fine natural aggregate with FRA in sodium or magnesium sulfate solutions, showing lower expansion. Moreover, RAMs with 75 and 100% show higher damage than control mortar. On the contrary, Santillán et al. [69] report higher damage for mortar with 30% FRA than for mortar with 50% FRA, in terms of expansion and weight variation. The different experimental setups of each study can explain this difference between them. For example, no water correction for FRA absorption is reported in Lee [59] or Lee et al. [60], and so the effective w/c ratio could be lower for low replacement values. On the other hand, in Santillan et al. [69], additional water was added to the dosage to compensate for FRA absorption. However, the determination of this value is not accurate and involves significant error [137].

Although there are few results in the literature, they show a predominant effect of the quality of the RAM matrix on their sulfate resistance, as in the case of RAC. Boudali et al. [91] and Candamano et al. [94] evaluated RAM with higher strength levels (self-compacting) and 100% FRA. The results show similar or even better performance for RAM than for the reference, especially when using an SCM in the composition.

As with RAC, RAM performance shows a possible positive effect of FRA against ESA, which, depending on other parameters, can compensate for the negative effect of the higher porosity of this aggregate.

3.2. ESA + PSA

Few studies have evaluated the performance of RAC under exposure conditions that promote PSA. Figure 3 shows the results collected from the literature.

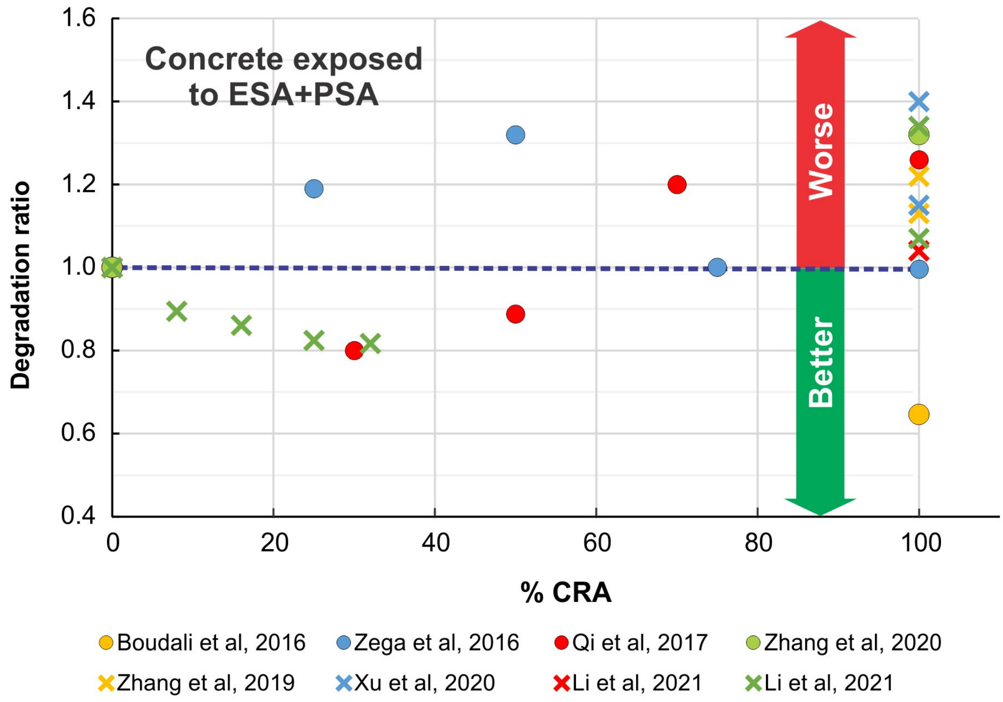

Figure 3. Degradation ratios of concretes with different % CRA exposed to ESA + PSA [63,65,77,83,84,88,89,91].

Most of the cited works compare conventional concretes to concretes with 100% RA [55,63,77,84,89,91]. Most results show worse performance for RAC than for NAC under this type of exposure, with the degradation ratio ranging from 1.04 to 1.39. The only case where better results were obtained was in Boudali et al. [91], where self-compacting concretes with 100% RA and different cementitious compositions (replacement by SCM) were studied. In this case, significantly lower degradation ratios were observed in the RAC than in the standard, with the SCM having a significant effect.

Only three papers evaluate the performance of concretes with partial RA replacement, namely, the studies by Zega et al. [83], Qi et al. [65], and Al-Baghdadi [68]. As described for ESA, the results do not show a consistent trend in the relationship between RA content and deterioration, which can be attributed to the different setups of each campaign. In addition, better performance of RAC is sometimes observed with respect to the reference concrete, again showing the possible positive effect of RA in some cases.

In Zega et al. [83], two series of concretes were evaluated: Series 1 with w/c ratio = 0.50 and Series 2 with w/c ratio = 0.38. For the first series, after ten years of exposure to saline soil (semi-buried), the decrease in the modulus of elasticity of the concretes with 25 and 75% was higher than that of the reference concrete. On the other hand, for the second series, practically no differences were observed between the NAC and the RAC, showing that the matrix quality can control the possible adverse effect of the RA.

RAC tested by Qi et al. [65] was subjected to wetting and drying cycles in a sodium sulfate solution. In this case, smaller decreases in the modulus of elasticity of RAC than those of the reference concretes were observed for those made with 30 and 50% RA and larger decreases for concretes with 70 and 100% RA. These results are confirmed by the sulfate ingress profiles calculated by analytical chemistry. The authors attribute this result to a slight change in the effective w/c ratio due to the moisture state of the aggregates.

Finally, Al-Baghdadi [68] used concretes made with RA pretreated by saturation in a polyvinyl alcohol (PVA) solution and subjected to saturation and drying cycles with a magnesium sulfate solution. In this case, the concretes obtained better results as the inclusion of RA increased, and the authors attribute these results to an internal curing effect generated by the RA and promoted by the addition of PVA.

As in the case of ESA, the results regarding ESA + PSA are not enough to establish consistent correlations, but they do confirm some points regarding the development of the sulfate attack in recycled concretes, such as the quality of the new matrix, the ambiguous effect of RA, and the very positive effect of SCM with RA.

3.3. ISA

As mentioned above, the ISA issue becomes relevant for RA due to the potential for contamination with sulfate-rich building materials, mainly gypsum. Most regulations prescribe the limiting content of sulfates in aggregates as a total percentage of their weight, with values between 0.2 and 1.0% [9,11]. However, several studies have investigated concretes and mortars with recycled aggregates contaminated with sulfate-based materials, and the results show that these limits may be conservative.

A summary of the reviewed papers is presented in Table 1. Several studies evaluate the effect of the sulfate content in RA [46,48,73,80,81]. For example, Tovar-Rodriguez et al. [73] show that mortars with 4.3% SO_3 content present three to four times greater expansion than mortars with 2.9% SO_3 content, even when an SRPC is used. The authors also demonstrate that 100% FRA mortar performs adequately in terms of durability, even with a 2.9% SO_3 content, which exceeds regulatory limitations. They base this conclusion on a mathematical prediction of service-life expansion. Agrela et al. [46] investigate samples of cement-treated granular material (low cement content and mechanical compaction) with 100% RA, different cement types, and different levels of gypsum contamination. They show that by using an SRPC, the SO_3 content of the aggregate can be increased up to 1.3% without any risk of ISA failure.

Table 1. Summary of papers on ISA in RAC.

Paper	Mixes	Sulfate	Highlights
Tovar-Rodriguez et al. [73]	RAM w/c = 0.35 100% FRA OPC and SRPC	FRA with SO_3 = 2.9% a.s. Contamination with different sulfate sources to increase SO_3 content to 4.3% bwa.	• Mortars with 100% of FRA with 2.9% SO_3 perform well in terms of durability (higher than regulation limits). • SO_3 bwa content of the RAMs has a much higher influence than the cement sulfate resistance.
Agrela et al. [46]	CTGM 100% CRA OPC and SRPC	CRA with SO_3 = 0.3% a.s. Contamination with hydrated gypsum to increase SO_3 content to 1.0 and 2.5% bwa.	• The limiting SO_3 content of the aggregate can be increased if SRPC is used in the mix (up to 1.3%).
Colman [47]	RAM w/c = 0.35 to 0.65 100% FRA OPC and SRPC	FRA with SO_4 = 0.18% w.s. Contamination with different size gypsum grains to increase SO_4 content to 0.47% and 3.08% bwa.	• The expansion of RAMs increases with SO_3 content. • Other set parameters, such as RA temperature or alkalinity, have a noticeable effect on RAMs' behavior.
Abid et al. [45]	RAC w/c = 0.55 0 to 100% CRA	CRA contaminated with laboratory-made plaster. SO_3 content not reported.	• Greater expansion occurs in concretes with higher CRA content.
Colman et al. [48]	RAM 0 and 100% FRA	RAMs with NA and FRAs with SO_3 = 0.08% to 0.62%. Contamination with gypsum increases the SO_3 content to 0.47% and 3.08% bwa.	• The FRA mortars perform similarly to the control mortar despite their SO_3 content of up to 0.68%. • The mortar contaminated with more than 3% SO_3 show a significantly higher degree of expansion.

Table 1. *Cont.*

Paper	Mixes	Sulfate	Highlights
Yammine et al. [81]	RAM (heat curing) w/c = 0.50 0 and 100% FRA OPC	Mortars with NA and two FRAs with SO_3 = 0.54% and 0.65% a.s., respectively. Contamination with sodium sulfate increases the SO_3 content to 5% bwc, and contamination with plaster powder increases the SO_3 content to 0.80% and 1.20% bwa.	• Among contaminated mortars, better performance is reported for mortars with FRA than for the reference mortar. • Mortars in which FRA is contaminated with 1.2% SO_3 show similar expansion to the control.
Yammine et al. [80]	RAC (heat curing) w/c = 0.50 0 and 100% CRA OPC and SCM	Concretes with NA and two CRAs with SO_4 = 0.11% and 0.29% w.s., respectively. Contamination with sodium sulfate increases the SO_4 content to 2.7% bwc.	• Better performance is observed in concretes with recycled aggregate than in reference concrete.

CTGM: cement-treated granular material; a.s.: acid-soluble; w.s.: water-soluble; bwa: by weight of aggregate; bwc: by weight of cement.

Colman et al. [48] evaluate mortars with contaminated FRA and different set-up parameters, such as temperature, alkalinity, and gypsum source. Mortars with higher SO_3 content showed higher expansion, but other parameters showed a considerable effect, such as temperature, sulfate resistance of cement, and alkalinity (due to CH lixiviation of RAs). Colman et al. [47] evaluate RAMs using several commercial FRAs with sulfate contents ranging from 0.08% to 0.62% and mixtures contaminated with gypsum (up to 3.08% SO_3). Mortars with commercial FRAs showed similar behavior to control mortar with NA, while mortar with 3.08% SO_3 showed considerably greater expansion. The authors suggest that some regulations may have conservative prescriptions (0.2% in the authors' location).

Other studies have shown the effects of RA on the development of ISA. Yammine et al. [81] evaluate mortars with and without RA from two different sources contaminated with sodium sulfate. These mortars were subjected to temperature curing, which is common in the precast industry and may promote the subsequent development of DEF (delayed ettringite formation). In this case, slightly greater expansion was found in the uncontaminated samples for the FRA mortars. At the same time, a strong positive effect of using FRA was observed in the contaminated samples. Microstructural analyses showed that the mortars with FRA had greater incorporation of air and, consequently, a higher number of small air bubbles (20 to 200 µm) acting as crystallization points without associated confinement. Once again, these results highlight the ambiguous effect of porosity on deterioration processes involving the formation of expansion products [100,142,143]. For example, Colman et al. [47] show greater expansion for mortar with a lower w/c ratio and, hence, lower porosity. Yammine et al. [80] evaluate the expansion of NAC and RAC cured at elevated temperatures. They report lower expansion for RAC than for NAC, which is attributed to the lower internal constraint that can be achieved using recycled aggregates. Moreover, the results of some papers [47,80] show that higher alkalinity for RA can improve the performance of mixes against the ISA, and it should be considered in further experimental setups. In the case of Abid et al. [45], concretes with contaminated RAC are evaluated for ISA. The results show an increased loss of mechanical strength with increasing RA content. However, only one level of sulfate contamination was used (not reported well), and so it cannot be confirmed whether the increased deterioration is an effect of the RA or the associated increased gypsum contamination.

Studies show that the aggregate sulfate limits set by regulations may be conservative. However, more experimental results are needed to better understand the effects of RA on the ISA under different conditions.

4. Discussion

4.1. Effect of RA

Our literature review shows that RAs can affect the performance of cementitious mixtures exposed to sulfate attack. The general trend shows worse performance in RACs than in NACs, mainly due to a higher porosity that accelerates the kinetics of the degradation mechanisms. Several hypotheses attempt to explain this phenomenon. Some authors point out that the higher porosity of RAC results in a lower internal restraint to deformation (lower modulus of elasticity), which delays crack propagation initiation [62,65,67,94]. The better strain compatibility of RA than NA with the cement matrix has previously been reported by Corinaldesi et al. [131]. Yammine et al. [81] attribute the lower constraint to the higher amount of entrapped air in RAC mixes [126], which creates mesopores that can act as preferential unconfined crystallization spaces for the ESA products. Another less consistent hypothesis is that the expansive products can precipitate in the pores of the RA, resulting in lower internal stresses [60]. However, there is no conclusive evidence that ESA products precipitate preferentially in the pores of the RAC. Regardless of which hypothesis is correct, the main difficulty with this effect is that it follows different patterns for different experimental campaigns. This suggests that other test parameters also influence the relationship between the negative and positive effects of porosity (e.g., materials used, exposure conditions, evaluation method).

The results show that in most cases, the performance of RAC depends more on the physical–chemical quality of the new matrix than on the quantity of RA included. In many works, an almost null effect of RA is observed in mixes with a low w/c ratio or with SR-cement, even for large replacements with RA. Instead, the effects of RA are observed most in concretes of lower quality. An important aspect related to porosity is that RA can modify the effective w/c ratio in its surroundings if the moisture compensation during the mix design and elaboration stages is incorrect [23,40,136,137,144]. This can lead to experimental errors, even with partial replacement by RA, as we would be adding or removing free water to or from the concrete during its hydration process. This difficulty is more significant in the case of FRA due to the difficulty of measuring water absorption in this fraction [145].

For ESA, studies have not yet evaluated the effects of the physical–chemical quality of the RA. The number of studies is very small, and the characterization of the RA is insufficient to draw sound conclusions. The two most important properties of RA that may affect ESA mechanisms are the porosity and the reactive phase content in its cement paste (AFm, CH, and C-S-H). The porosity of RA depends, as mentioned above, on the mortar/paste content, the quality of the original concrete, and the crushing process, among other factors. The mineral composition of the RA, specifically the cement paste, depends on the type of cement in the source concrete and the degree of carbonation of the RA. For ISA, the mineral composition is also strongly influenced by contamination, particularly sulfate-based materials.

The characterization of RA must be accompanied by a study of their effect on RAC. It is interesting to know whether RA's potential reactivity becomes effective under certain exposure conditions or under different design criteria. Further investigations are needed to determine whether the contribution of reactive phases may have an additional effect. In addition, knowledge about the ambiguous effect of RA observed in several studies should be deepened. The results achieved in relation to the aforementioned premises can lead to accurate classification protocols for RA and design specifications for recycled mixes.

4.2. Sulfate-Resistant RAC Design

Although the results analyzed are insufficient to fully explain the effect of RA on sulfate performance, they provide information about technological solutions that may improve the performance of RAC against sulfates. The literature review highlights the significance of the RAC matrix on the potential effects of RA. The results suggest that recycled concretes, designed using conventional concrete criteria (low w/c, SRPC), exhibit satisfactory performance even with substantial aggregate replacements. The discussion regarding admissible percentages in regulations and the potential impact of RA properties on these limits requires further research to draw reliable conclusions.

Another important discussion is about the limitation of sulfate content by RA to avoid the development of ISA. Existing studies indicate that the current limits established for natural aggregates are quite conservative. The existing limits for SO_3 content in aggregates, which range between 0.2% and 1.0%, could be further increased, especially if additional design criteria, such as the use of SRPC, are adopted. However, due to the scarcity of literature, reliable values for regulation cannot be defined.

In addition, many studies provide strategies to optimize the performance of RAC, among which three groups can be highlighted: the use of SCMs, RAC procedural improvements, and pretreatment of RA. The use of pozzolanic SCMs as a strategy to increase the resistance of concrete to sulfate attack has shown equal or even superior efficiency in RACs compared to their respective conventional concretes (NACs) [45,54,62,65,66,71,72,78,90,91,146,147]. This is extremely important because it means that eco-efficient concretes (such as those with RA and a lower clinker factor) can perform as well as or better than comparable conventional concretes in terms of resistance to sulfate attack. This synergistic combination of RA and SCM has been highlighted by several authors and linked to other concrete properties [89,127,130,138,148,149].

Some authors propose improvements in RAC through strategies in the design and processing procedure. For example, the two-stage mixing approach improves the mechanical properties of RAC, mainly explained by a substantial improvement in the ITZ [130,138,150], a fundamental phase in the transport properties of hardened concrete [36,138]. Other works point to design strategies. Bui et al. [151] suggest using only a fraction of the RA (the coarser fraction), allowing the replacement percentages to be increased without losing performance.

Finally, many studies evaluate different pretreatment methods for RA to improve overall durability performance [15,152,153], including resistance to sulfate attack [56,68,70,154]. Existing pretreatment techniques for RA are divided into attached-mortar improvement techniques and attached-mortar removal techniques [153]. Among the first, different cementitious slurries were tested by Shayan et al. [70] and Zhang et al. [154], showing a positive effect on RAC sulfate resistance. In another case, Kazmi et al. [56] evaluated the sulfate resistance of mixes containing RA pretreated by accelerated carbonation, highlighting the improvement in the quality of RA and the recapture of CO_2 from the atmosphere [22,56]. Al-Baghdidi et al. [68] evaluated the effect of pretreatment with a polyvinyl alcohol (PVA) solution. The results showed an increase in RAC sulfate resistance with increasing RA content. In this case, the PVA impregnation acted as a water reservoir, providing the conditions for internal curing. This potential characteristic of RA as an internal curing agent has also been studied with positive results and is a path to be developed [136,138].

4.3. Guidelines for Further Research

Based on the information collected and the analysis carried out, general guidelines are proposed for further research to fully understand the effect of RA on the sulfate-attack resistance of RAC. Table 2 summarizes the key points of this section.

Firstly, RA must be rigorously characterized at the physical and chemical level to assess their hygroscopic and thermodynamic behavior correctly [135,137,155]. The most suitable parameters for standardization are mortar content (cement paste for FRA), porosity, absorption, and paste composition (chemical and mineralogical) [44,134,156,157].

Regarding mixture design, different levels and qualities of RAC should be considered. For RAC performance testing, standard criteria should be established for matrix quality, evaluation methods, and exposure conditions. Matrix quality should be approached by varying the w/c ratio, changing the type of cement, using SCMs (different types and contents), and considering different mixture designs. In terms of measurement, in addition to the traditional methods used (nondestructive and destructive tests), the proposal of new methods or methods adapted from other tests may be necessary. The most useful approach is that RACs should comply with regulations' prescriptions for conventional concretes exposed to the same conditions, which are usually focused on a low w/c ratio and the use of SR binder.

Table 2. Test parameters to consider in further experimental campaigns.

Level	Group	Variables
Aggregate characterization	Physical properties	• Porosity • Absorption • Attached mortar/paste content
	Chemical properties	• Phase quantification (CH, AFm) • Sulfate content (SO_3) • Alkalinity
Performance tests on concrete and mortar samples	Matrix-related parameters	• Matrix porosity (w/c ratio) • Cement type (SRPC and others), SCMs, and admixtures
	Aggregate related parameters	• RA replacement • RA's physical and chemical properties
	Exposure related parameters	• ISA: sulfate contamination, alkalinity • Saturation condition (ESA or ESA + PSA), sulfate media concentration

In terms of exposure, accelerated tests are usually used, the concentrations of which generally fall within the most aggressive conditions of the regulations. In the case of saturated condition tests, this criterion is more justified, as diffusion transport makes ESA a process of prolonged deterioration over time. In the case of wetting–drying cycle tests, some works in the literature evaluate protocols and concentrations that provide better test times and have some correlation with long-term tests [117,158]. For partial saturation tests, there is no single criterion, but in general, these tests use semi-buried or semi-submerged specimens in soils or sulfate solutions, with concentrations that are also very aggressive [57,88,159]. Regardless of the method applied, the most relevant aspect to delve into is finding information that includes the results of accelerated tests and field exposures.

In the case of ISA, the published results show that the regulations should consider the case of RA, especially regarding the limited content of soluble SO_3. Further studies can be carried out to better adjust the allowable limit according to other parameters of the mix, such as the type of cement, alkalinity, and strength level of the new material. It is also interesting to know the effect of temperature curing [85,86], which is a common practice in the precast industry. In addition, the effect of different types of contaminants on the RA should be evaluated [55].

Finally, the potential of simulating degradation processes by formulating numerical mathematical models [96,160–162] is worth mentioning. The main advantage of these methods is that they significantly reduce the logistical and operational costs of testing, especially when many variables need to be evaluated, as in the case of sulfate attack. However, the numerical models themselves require traditional tests to be calibrated. This area of research is still under development, but its potential is very promising.

5. Conclusions

This review summarizes the performance of cementitious mixture with recycled concrete aggregates (RA) against different types of sulfate attack. Existing studies about this topic were compiled, and their results were analyzed in terms of degradation ratio and RAC content. Based on the above, the following conclusions can be drawn:

- The performance of RAC against sulfate attack is strongly dependent on the properties of the new cement matrix, with well-designed recycled concretes demonstrating good performance even with substantial amounts of RA as replacements.

- Using RA generally decreases concrete's resistance to sulfate attack due to increased transport properties. However, in some cases, RA can mitigate the negative effects by compensating for increased porosity.
- It remains unclear whether RA can significantly increase the quantity of sulfate-reactive phases, necessitating further studies with detailed characterization of RA and concrete performance tests using the same RA.
- The combination of RA with SCM can have a certain synergy of positive effects, resulting in even higher eco-efficiency for the material. Various pretreatment techniques for RA can also increase the sulfate resistance of RAC.
- The limits for SO_3 regulations should be adjusted for recycled aggregate, as the current limits for natural aggregates may be inadequate for RA. These limits could be even higher if special precautions are taken in concrete design, such as using SR cement or SCM.

Further research should explore aspects such as internal restraint and potential reactivity of RA. The development of a standard method, including test conditions and evaluation parameters, would greatly benefit the study of different types, qualities, and quantities of RA in cementitious mixtures.

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Nomenclature

CDW	construction and demolition waste	ESA	external sulfate attack
RA	recycled concrete aggregates	ISA	internal sulfate attack
RAC	recycled aggregate concrete	PSA	physical sulfate attack
RAM	recycled aggregate mortar	CH	calcium hydroxide
NA	natural aggregates	SCM	supplementary cementitious materials
NAC	natural aggregate concrete	ITZ	interfacial transition zone
CRA	Coarse Recycled Aggregate	OPC	Ordinary Portland Cement
FRA	Fine Recycled Aggregate	SRPC	Sulfate-resistant Portland

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