Expansion of concrete made with fine recycled aggregates contaminated with gypsum

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Abstract

Recycled aggregates, and especially the fine (0/4 mm) fraction, are often contaminated with sulfates coming from gypsum residues on the demolition site. When these aggregates are used in concrete, the sulfates can induce internal sulfate attack which causes the expansion of concrete. Standard EN206 sets the water soluble sulfate limit at 0.2% by weight of the aggregate but other studies suggest this limit could be safely increased. In addition to the sulfate content, other parameters like the porosity and alkalinity of a mix have been seen to influence the swelling results. In this study, the different proposed sulfate limits are evaluated on concrete made with recycled aggregates. It is also researched whether mixing parameters could change the swelling amount regardless of sulfate content. The results showed that the incorporation of fine recycled aggregates with sulfate contents up to 0.8 mass% is safe when combined with coarse natural aggregates. If coarse recycled aggregates are used, the sulfate content of fine recycled aggregates could reach up to 0.3%.

Keywords: Recycled aggregates, sulfate attack, waste management, secondary ettringite formation, expansion

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1. Introduction

The construction industry is one of the most energy and resource consuming sectors in the world and produces an enormous amount of construction and demolition waste (C&DW) [1]. This C&DW consists mostly of crushed concrete [2] and the challenge exists in valorizing this waste stream. C&DW can be reprocessed into recycled aggregates, that can be used inside a new concrete structure as a replacement for natural aggregates [3, 4]. Using recycled aggregates is a practice that decreases the environmental impact of the construction sector by reducing the need for landfills, aggregate extraction and transport [5]. 1.7 tonnes of these recycled aggregates are produced per person per year in Europe, waiting to be valorized [6].

While coarse recycled aggregates (CRA) are already used in various applications without important losses in properties [7, 8, 9, 10, 11, 12], fine recycled aggregates (FRA) are up to now not valorized [13, 14]. Their higher water absorption [15, 16], lower density, and the presence of contaminations from the construction or demolition site such as plaster, bricks, wood, ... [17, 18] are cited among the reasons why.

Gypsum ($CaSO_4.2H_2O$) is often found as a contamination in recycled aggregates, which could have originated from different sources. The demolition of a building will cause gypsum residues - coming from plaster and drywall - to end up in FRA. Gypsum is also used as an addition to Portland cement, to regulate its setting time and prevent a flash set [19]. These gypsum residues are more problematic for the finer size fractions of recycled aggregates, because larger concrete and gypsum particles can still be separated from each other based on a difference in color [20] or density [21], and because the FRA contain a high residual cement content [22, 23, 24]. There is a clear variability between different recycling centers: in industrial FRA samples, values of 0.03-0.25% [25], 0.15-0.8% [26] and up to 1.52% [27] of sulfates have been found in different studies. The valorization of FRA is strongly limited by contaminations with water soluble sulfates [28], because they can induce internal sulfate attack.

Sulfate attack is a deteriorating process for concrete where sulfates react with water and aluminates from cement to form ettringite [29, 30]. Primary ettringite is a normal hydration product in the cement paste: it is only secondary ettringite, formed in an already rigid cement matrix, that risks deteriorating the concrete [31]. Ettringite is an expansive mineral, and will exert a pressure on its surrounding cement paste [32, 33, 34]. The volumetric deformation caused by this reaction can in its turn induce (micro)cracking [19] and a general loss in mechanical performances. Macroscopically, the swelling of concrete can be measured as an indication of internal sulfate attack.

A distinction can be made between different types of sulfate attack. External sulfate attack happens when the sulfates diffuse into the concrete from an aggressive environment [34]. Another reaction called Delayed Ettringite Formation (DEF) occurs when primary ettringite is destroyed by high curing temperatures, and formed anew in a hardened cement paste. The sulfates in this case come from cement, an internal source.

While external sulfate attack and Delayed Ettringite Formation are known reations, but the context where swelling is caused by the presence of gypsum in FRA is not well researched. The gypsum residues contaminating FRA are another internal source of sulfates and unlike with the DEF reaction, high curing temperatures are not needed to observe the swelling effect of ettringite formation. The term 'secondary ettringite formation' will be used to distinguish this reaction from DEF.

To keep the risk for secondary ettringite formation at a reasonable level, the current water soluble sulfate limit in coarse recycled aggregates is established at 0.2% by EN206 [35], with no specific mention of FRA. The conclusions of recent durability studies indicate a higher level should be made possible [36], specifically up to contents of 0.3% [37].

Research on mortar samples made with FRA and an elevated sulfate concentration of 3% showed that several mixing parameters could influence the swelling results without changing the contamination level itself. Two notable parameters were discovered: a limited porosity enhanced the total expansion, and an increased alkalinity inhibited it [38]. The most commonly accepted theory about the cause of expansion is the heterogeneous crystal pressure exerted by the growing ettringite crystals. In this sense, a lower porosity means more confinement and a higher internal pressure. The alkalinity of the interstitial solution interferes with the equilibrium between the different sulfate phases. A higher alkalinity favors the existence of monosulfate and the absorption of sulfur on the C-S-H gel instead of the formation of ettringite [39], so ettringite formation will trigger as pH lowers. Next to a decrease in swelling results, a higher mechanical performance was found too: alkalinity speeds up hydration and increases early compressive strength [40].

In this study, the effect of sulfate concentration of FRA on the swelling reaction has been analyzed to evaluate different (proposed) sulfate limits. Next, the relevant parameters that could influence the swelling results are studied: can an allowed sulfate content still cause swelling because of a limited porosity, and can an increased alkalinity mitigate the swelling caused by an otherwise rejected sulfate content?

2. Materials and methods

2.1. Used materials

Recycled aggregates were produced in the laboratory by fabricating a standard concrete and subsequently crushing it. The use of 'model' recycled aggregates gave exact control of the chemical composition of the materials and removed any possible variability or contamination at the level of the aggregates by chlorides, organics, etc. The composition of this original concrete is given in Table 1: it was designed to obtain a consistency class S3 and strength class C30/37.

After 90 days of curing, this concrete was crushed by a jaw crusher and divided in two groups: fine recycled aggregates (FRA) of 0/4 mm, and coarse recycled aggregates (CRA) of 4/16 mm. Their size distributions are shown in Figure 1.

CEM I 52.5 N	Water	Limestone aggregates (mm)				Superplasticizer
		0/4	2/7	7/14	14/20	
350	175	216	658	436	612	0.4%

Table 1: Composition, in kg, of the original concrete

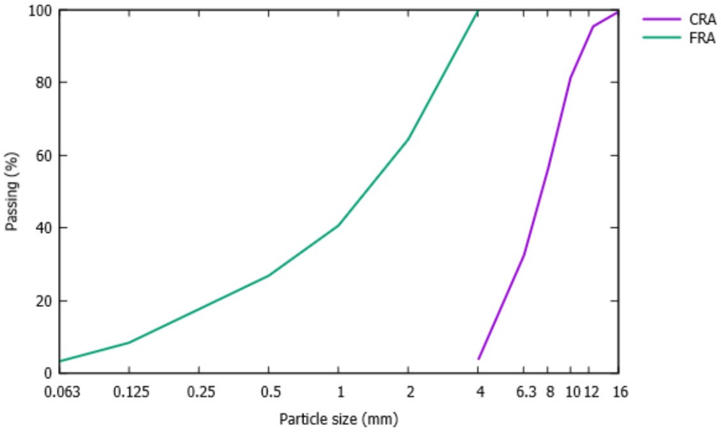

Figure 1: Size distribution of the used recycled aggregates.

	Water absorption (%)	Particle density g/cm^3	SO_4^{2-} content (%)
FRA (0/4 mm)	9.78	1.95	0.18
CRA (4/16 mm)	3.12	2.38	0.05
CNA (4/16 mm)	1.4	2.77	0

Table 2: Properties of the used aggregates.

While all concrete formulations contained *fine* recycled aggregates, the used *coarse* aggregates were either the CRA or natural limestone aggregates (CNA). These CNA were recomposed to have the same size distribution as CRA, so they resemble the model material. The properties of these 3 aggregate types - FRA, CRA and CNA - are shown in Table 2. The water soluble sulfate content was determined via leaching and analysis with ion chromatography [41]. The water absorption and density of recycled materials was measured with the method described by Zhao et al. [15].

Depending on the concrete formulation, the 0/4 mm fraction of the recycled aggregates was manually contaminated with gypsum - a $CaSO_4.2H_2O$ powder (D50 13 μm) obtained from VWR Chemicals. A CEM I 52.5 N cement from HOLCIM was used; its chemical composition is shown in Table 3. To reach the desired slump, the ViskoCrete superplastifier from Sika was added to the concrete during mixing.

Chemical	CEM I 52.5 N
CaO	64.3
SiO_2	18.3
Al_2O_3	5.2
Fe_2O_3	4.0
MgO	1.4
Na_2O	0.32
K_2O	0.43
SO_3	3.5
Cl^-	0.06
LOI	2.3
C_3A	6.6
C_4AF	12
C_3S	61.9
C_2S	11.2

Table 3: Chemical composition (mass%) of the cement.

2.2. Concrete fabrication

Following previous research, 4 series of experiments were envisaged:

- The sulfate limit was evaluated on concrete with FRA and CRA. Different amounts of gypsum contaminations in the FRA were used.
- These same gypsum contaminations were tested on concrete with FRA and CNA.
- A limited porosity, which was found to be an aggravating parameter, was tested on a concrete that contained the maximum sulfate limit according to EN206.
- An increased alkalinity, which was found to limit the swelling amount, was tested on a concrete with a sulfate content that is normally not accepted.

The nomenclature of the mixes is as follows: [natural or recycled coarse aggregate] - [W/C ratio] - [sulfate content as a mass% of FRA] (-[added alkalinity]).

The design of the concretes was done with the Dreux-Gorisse method: the resulting compositions are given in Table 4. Because the aggregate envelope volume was kept constant, the amounts of CRA and CNA depended on their density. To compensate for the elevated water absorption of recycled aggregates, they were presaturated with their absorbed water and 15% of the mixing water, one week before mixing. Presaturating recycled aggregates has been shown to improve the maniability of a mix [42].

The mixing protocol is given in Table 5. The presaturated aggregates were placed in the mixer after which the water, cement and superplastifier were added. The superplastifier was added progressively and the slump of the concrete was checked with the help of an Abrams cone according to EN 12350-8, until a value of 10 to 15 cm was obtained. The fresh properties of the concrete mixes are shown in Table 6. 7x7x28 cm bars with measuring pins were cast for weekly swelling tests, and 15x15x15 cm cubes for the periodic mechanical measurements.

Name	FRA	CRA	CNA	Cement	Water	Superplast.	Gypsum	NaOH
R-0.5-3.1	15.2	28.2	0	10.5	7.62	0.0355	0.785	
R-0.5-0.8	15.8	28.2	0	10.5	7.68	0.0317	0.148	
R-0.5-0.3	16.0	28.2	0	10.5	7.69	0.0207	0.033	
R-0.5-0.2	16.0	28.2	0	10.5	7.69	0.0216	0.005	
N-0.5-3.1	15.2	0	32.8	10.5	7.20	0.0355	0.806	
N-0.5-0.8	15.8	0	32.8	10.5	7.26	0.0236	0.171	
N-0.5-0.3	16.0	0	32.8	10.5	7.27	0.0456	0.033	
N-0.5-0.2	16.0	0	32.8	10.5	7.27	0.0121	0.005	
N-0.35-0.2	16.0	0	32.8	10.5	5.70	0.1104	0.005	
N-0.5-0.3-A	16.0	0	32.8	10.5	7.27	0.0431	0.033	0.0224

Table 4: Compositions, in kg, of the concrete mixes.

Action	Time	Total time
Add aggregates (0/16 mm), mix	30”	30”
Add half of the mixing water, mix	2’	2’30”
Rest	2’	4’30”
Add cement, mix	30”	5’
Add second half of mixing water and superplastifier, mix	1’30	6’30”

Table 5: Concrete mixing procedure in function of mixer speed.

Name	Slump (cm)	Density (g/cm^3)
R-0.5-3.1	12	1.894
R-0.5-0.8	14.5	2.066
R-0.5-0.3	10.5	1.958
R-0.5-0.2	14.5	1.726
N-0.5-3.1	11	2.335
N-0.5-0.8	11	2.007
N-0.5-0.3	11.5	1.918
N-0.5-0.2	11.5	1.948
N-0.35-0.2	15	2.079
N-0.5-0.3-A	15	2.075

Table 6: Fresh properties of the concrete mixes.

2.3. Monitoring of the reaction

To follow the development of the internal sulfate attack reaction, the concrete specimens were subjected to different tests. The length change of the concrete bars was recorded weekly with a digital length comparator, in reference to an Invar bar. At 7, 28 and 90 days the concrete samples were characterized mechanically for their compressive strength [43]. Every described test was done for 3 replicates.

3. Results and discussion

In a first series of experiments, the sulfate limits were evaluated. Standard EN206 sets this limit at 0.2%, while the French national project RecyBéton proposes to increase this to 0.3%. Two other high sulfate contents, 0.8% and 3.1% were tested too.

In Figure 2 the swelling results of the concretes made with FRA and CRA can be seen, and in Figure 3 the results for concrete made with FRA and CNA. For the "R" series made with recycled aggregates, two groups of results can be noticed: those with elevated sulfate contents indeed showed an important swelling, but the mixes with 0.2 and 0.3% of sulfates did not swell significantly. This indicates that the proposed increase to 0.3% is feasible. While no difference in swelling results could be seen between 0.2 and 0.3 % of sulfates, the compressive strength did show a difference. The concretes with the lowest and highest amount of sulfates performed worse than those with intermediate levels. It seemed that adding a little gypsum improved the compressive strength, but that 3.1 % was already too much to have a beneficial effect on compressive strength.

For the "N" concretes which contained CNA in addition to FRA, the mix with 0.8 % of sulfates also showed an acceptable expansion together with the two lower contamination levels. This means that depending on the concrete formulation, even higher amounts than 0.3 % must be possible. The use of CNA lowered the differences between the samples in compressive strength.

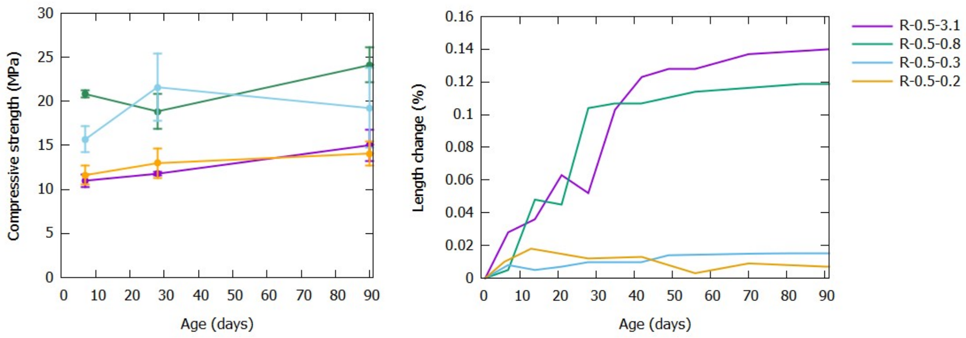

Figure 2: Compressive strength and swelling results for the concrete series with FRA and CRA and varying sulfate contaminations.

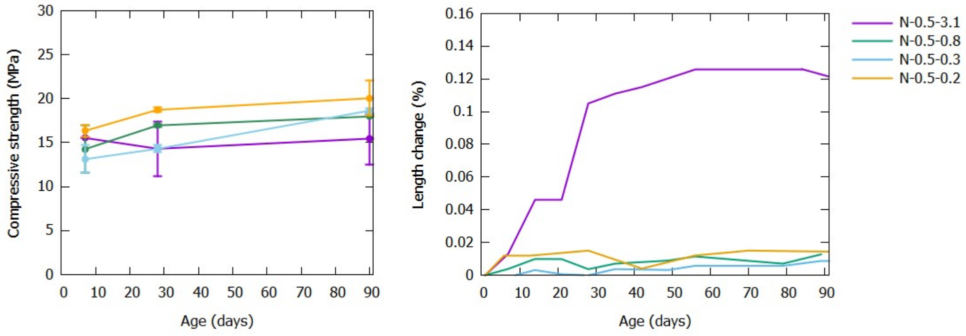

Figure 3: Compressive strength and swelling results for the concrete series with FRA and CNA and varying sulfate contaminations.

The mix with 3.1 % of sulfates reached the same expansion with either recycled or natural coarse aggregates. It seemed that as soon as swelling occurred, the absolute amount was always the same, unrelated to the sulfate content or the type of coarse aggregate. What *Collepardi et al.* [19] suggested about the necessity of microcracks could explain why there was almost no difference in swelling results between "R-0.5-3.1", "R-0.5-0.8" and "N-0.5-3.1", or all other concrete bars with lower sulfate levels. As soon as the swelling process starts it maintains and accelerates itself regardless of actual sulfate contents. There seemed to be a certain treshold of sulfates for when a concrete started swelling, and the use of CNA increased this treshold: the sulfate content needed to kick-start the expansion was between 0.3 and 0.8 for the "R" series and between 0.8 and 3.1 for the "N" series.

An explanation for this difference in swelling between "R-0.5-0.8" and "N-0.5-0.8" could lie in the pore size distribution. Figure 4 shows the volume of mercury intrusion for different pore sizes of these samples at 90 days. Concretes made with CNA showed two distinct groups of pores at 0.01-0.1 μm and 1-10 μm, while mixes with CRA only exhibited the smaller pore sizes at 0.01-0.1 μm. Ettringite crystals as a hydration product are in the 1-5 μm size range [**?**], and SEM images of massive ettringite deposits in deteriorated concrete show sizes from a few to 15 μm [44, 45]. It would make sense that these larger pore sizes get 'filled up' first and the "N-0.5-0.8" has enough reserve of these pore sizes to accommodate this.

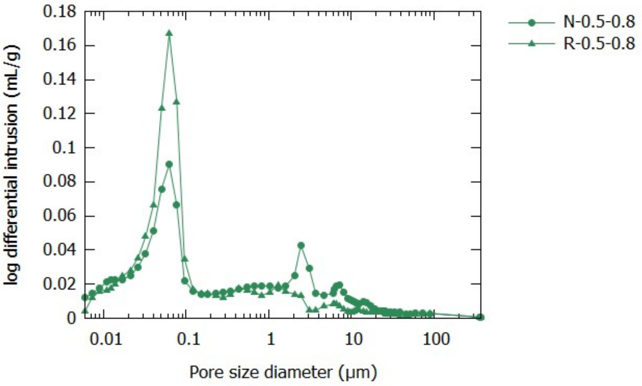

Figure 4: The pore size distribution at 90 days of R-0.5-0.8 and N-0.5-0.8.

The porosity of a mix, influenced by its W/C ratio, has been shown to be an enhancing factor for the expansion due to internal sulfate attack. For an acceptable sulfate content of 0.2%, this means that the expansion could still be significant with low W/C ratios. Figure 5 shows this was not the case. A sulfate contamination of 0.2 % was not enough to provoke a significant swelling reaction even in these aggravating circumstances. As expected, a low W/C did cause a higher compressive strength. The actual porosity of these mixes, measured by Mercury Intrusion, is presented in Figure 6.

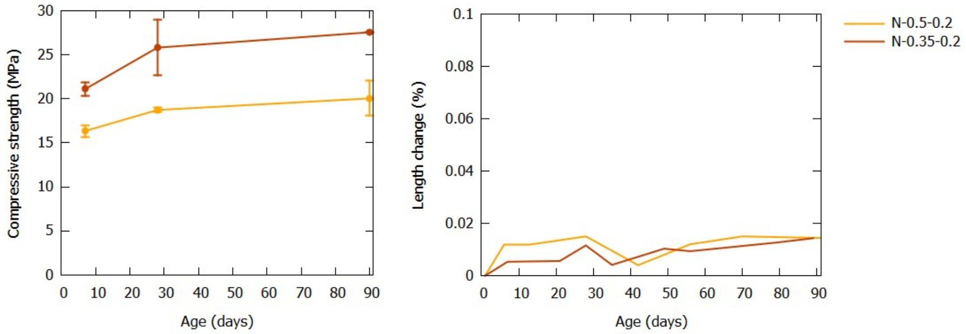

Figure 5: Compressive strength and swelling results for the concrete series with the maximum allowable sulfate content and a varying porosity.

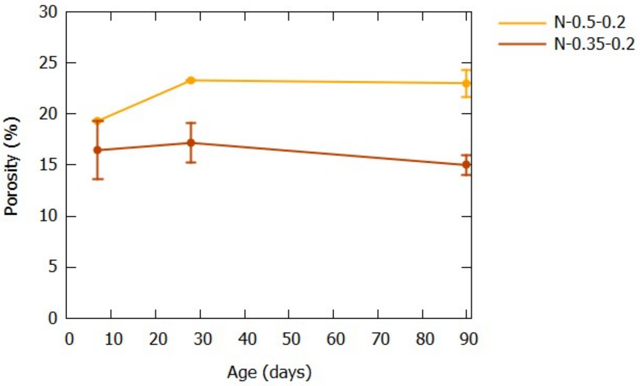

Figure 6: Porosity of the concrete series with the maximum allowable sulfate content and a varying porosity.

As seen in a previous study, increasing the alkalinity of a mix will inhibit the swelling effect caused by a sulfate contamination. A sulfate level of 0.3 %, which was proposed as a safe contamination by recent research but not yet accepted by international standards, was chosen as a reference. Although the expansion with 0.3 % of sulfates is not significant, an increased alkalinity still lowered this amount. The compressive strength of this mix was very high compared to the other concretes in these experiments. The increased performance caused by alkalinity was already described by other authors.

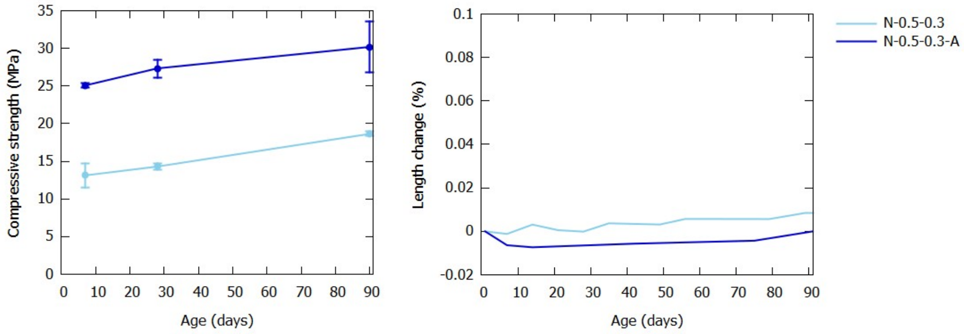

Figure 7: Compressive strength and swelling results for the concrete series with a higher sulfate content, and a varying alkalinity.

4. Conclusion

The sulfate limit was evaluated on concretes with fine recycled aggregates. Both the limit of 0.2% from EN206 and the proposed one of 0.3% proved to be safe and did not provoke any significant swelling. Depending on the nature of the coarse aggregates (natural or recycled), the sulfate level at which swelling starts may differ. For CRA, this concentration was between 0.3 and 0.8 mass% of FRA, and for CNA this was between 0.8 and 3.1 mass% of FRA. This difference in limits could be due to the pore size distribution: a lack of pores in the 1-10 μm range means less resistance to swelling. The amount of expansion did not correlate with the amount of sulfates: as soon as swelling started, the length changes stayed roughly the same regardless of sulfate contents.

These results showed that the sulfate limit of 0.2% could be seen as too strict, and that an increase to *at least* 0.3% should be possible. Unless a high contamination level is present, a limited porosity will not worsen the swelling, and an increased alkalinity would not be necessary to limit it.

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