Self-healing concrete with recycled aggregates

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Julia García-González[a], *Desirée Rodríguez-Robles*[b], *Nele De Belie*[c], *Julia Mª Morán-del Pozo*[a], *M. Ignacio Guerra-Romero*[a], *Andrés Juan-Valdés*[a]
[a]Department of Agricultural Engineering and Sciences, University of León, León, Spain, [b]Department of Agronomy and Forestry Engineering, University of Extremadura, Badajoz, Spain, [c]Magnel Laboratory for Concrete Research, Ghent University, Ghent, Belgium

18.1 Introduction

The overconsumption of resources and the elevated generation of wastes are two of the most relevant environmental challenges of the 21st century. Traditionally, natural resources and wastes are found, respectively, at the start and end point of the linear model of production, i.e., the material life cycle, which encourages the take-use-dispose attitude responsible for the problems in the first place. Nonetheless, in the recent years, a new circular economy model has been supported by scientific efforts, political attempts, and social awareness to transform the production chains and consumption habits by means of turning waste into resources.

In the path toward the sustainability in the construction sector, numerous studies have shown the feasibility of using recycled aggregates (RA) as replacement of natural aggregates (NA) for the manufacture of recycled concrete (Xuan et al., 2017; Thomas et al., 2018; Agrela et al., 2011; De Brito et al., 2005; Huda and Alam, 2014; Limbachiya et al., 2000; Poon et al., 2004a; Rahal, 2007; Rodríguez et al., 2016).

However, to date, the higher water absorption (WA) of recycled aggregates is still regarded as a major barrier in the reuse of this secondary material and thus limiting its acceptance in the construction market (Sagoe-Crentsil et al., 2001; Evangelista and de Brito, 2007; Poon et al., 2004b; Tam et al., 2005). Thus, further research on the improvement of RA properties and the subsequent study of the feasibility to use them as a replacement of the conventional aggregate in the manufacture of concrete is still needed.

Among the available techniques to palliate the drawback posed by the higher absorption of RA, it is worth mentioning the application of bacterially induced calcium carbonate precipitation as an eco-friendly technique to reduce the water uptake capacity of RA and thus to improve their quality to be reused in the concrete production. The biogenic $CaCO_3$ acts as superficial treatment, covering the external micro-cracks of the RA and filling some superficial pores, as well as some of the deeper ones (Grabiec et al., 2012; Qiu et al., 2014; García-González et al., 2017). Furthermore, calcium carbonate precipitation has been proven to increase the RA density and strengthen their surface (García-González et al., 2017; Wang et al., 2017), which positively affects the mechanical and durability performance of the resulting recycled concrete.

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18.2 Water absorption of recycled aggregates. Ways to solve the problem

It is well known that the water absorption of RA is significantly higher than NA. The reason behind this fact could arise from the adhered mortar remaining after the CDW treatment process or the intrinsic nature of the recycled material (e.g., bricks). Therefore, different techniques have been attempted to solve the water absorption issue and thus achieve an enhancement of the properties of RA for their use in the concrete manufacture.

On one hand, researches have attempted to circumvent the water absorption issue by different techniques focused on controlling the expected extra absorption of RA. Some studies simply contemplate the addition of extra water to the mixture in order to account for the predictable reduction of available water (Tabsh and Abdelfatah, 2009; Soares et al., 2014; Ferreira et al., 2011). For instance, it has been reported that the use of 100% coarse RA would involve, on average, up to 10% more water to achieve the same slump (Tabsh and Abdelfatah, 2009). However, the amount of water added would not be totally absorbed during mixing, thus leading to a higher water-cement ratio and lesser properties of concrete (Zhao et al., 2017). Other authors have recommended the use of recycled aggregates in a saturated state before their use in the concrete manufacture by means of the so-called presaturation technique (Ferreira et al., 2011; Zhao et al., 2017; García-González et al., 2014; Mefteh et al., 2013; Brand et al., 2015; Eckert and Oliveira, 2017). Nevertheless, a profound knowledge of the water absorption value and its evolution with time is a key to establish the precise soaking time and therefore the required amount of water to attain the degree of moisture is needed to eliminate the extra water capture that would originate due to the greater water absorption of RA without compromising effective water-cement ratio or the properties of the resulting recycled concrete (García-González et al., 2014). For instance, it was found that a 3-min presaturation and dry surface of mixed recycled aggregates led to workable mixes with a negligible decrease in the compressive strength compared to that of the conventional concrete (García-González et al., 2014). Finally, the use of water-reducing admixtures, such as plasticizers and superplasticizers (Barbudo et al., 2013; Matias et al., 2013; Bravo et al., 2017; Pereira et al., 2012) and also sugar cane molasses (Rashid et al., 2019) have been effectively used to palliate the water absorption drawback of RA. Furthermore, such practice has also led to achieve better workability and mechanical properties of recycled concrete (Barbudo et al., 2013; Matias et al., 2013; Bravo et al., 2017; Pereira et al., 2012; Rashid et al., 2019), although the improvement extent was dependent on the aggregate composition and on the property tested. Nonetheless, superplasticizers have been reported less effective in recycled concrete than in the conventional mixtures (Matias et al., 2013).

The second set of methods relates to the elimination of the adhered mortar which would constitute a new step in the CDW management. Pepe et al. (2014) suggested an autogenous cleaning process and reported a significant effect in the reduction of the water absorption (between 20% and 50%) after a 15 min process of self-attrition in a

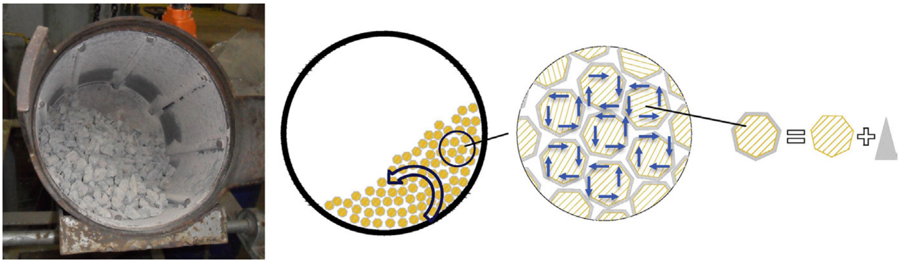


Fig. 18.1 Autogenous cleaning process (Pepe et al., 2014).

rotating (60 rpm) mill drum (Fig. 18.1). A decrease of 32.3% in the water absorption was also reported when RA was pretreated in a Los Angeles abrasion machine—300 revolutions with 12 charges—(Pandurangan et al., 2016). On a similar note, Dimitriou et al. (2018) employed a concrete mixer truck to pretreat the RA at 10 rpm for 5 h and recover > 4-mm cleaned RA after a washing and sieving process.

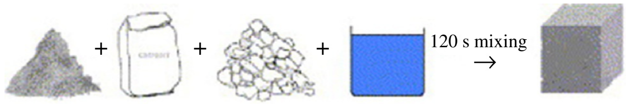
Several studies have focused on the use of an acid solution to remove the old mortar. A wide range of concentrations and immersion times, as well as different acids have been tested: hydrochloric acid (Tam et al., 2007a; Ismail and Ramli, 2014; Kim et al., 2018; Saravanakumar et al., 2016; Purushothaman et al., 2015), sulfuric acid (Tam et al., 2007a; Saravanakumar et al., 2016; Purushothaman et al., 2015), phosphoric acid (Tam et al., 2007a), and nitric acid (Pandurangan et al., 2016; Butler et al., 2011; de Juan and Gutiérrez, 2009), which has led to reductions between 7.27% (Tam et al., 2007a) and 36.7% (Pandurangan et al., 2016) in WA. Moreover, since a washing occurs after the soaking period, it has been demonstrated that neither the RA nor concrete are adversely affected by the pretreatment (Tam et al., 2007a). However, this method is unviable for limestone aggregates, since these would be attacked by the acid.

Some researchers have employed thermal shock treatments, i.e., heating in a furnace, 500°C for 2 h, and immediately submerging in cold water, to force the mortar detachment (Pandurangan et al., 2016; Butler et al., 2011). The method, which is normally followed by a minor mechanical degradation step, has proved to reach an almost complete mortar removal (Butler et al., 2011) and 17% water absorption reduction (Pandurangan et al., 2016). In addition, as previously described, mortar removal by microwave-assisted treatment has also been carried out (Bru et al., 2014; Akbarnezhad et al., 2011) as a less aggressive alternative to the thermal method. Among the thermal attempts, it is also worth mentioning the technique suggested by Abbas et al. (2008), who subjected RA to 5 freeze-thaw cycles, ranging between − 17°C and 80°C, while immersed in a sodium sulfate solution, which managed up to a 80%–90% removal rate (Butler et al., 2011).

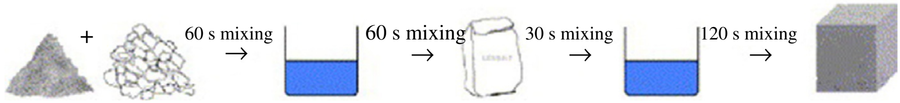
Lastly, also pertaining to the removal practices, ultrasonic cleaning has been used to detach the adhered mortar (Katz, 2004) which ultimately has led to a 7% improvement in the compressive strength of recycled concrete.

The third collection of techniques could be found in different approaches to the concrete mixing process designed to palliate the higher water absorption and lesser quality of the old mortar of RA. Tam et al. (2005) proposed the two-stage mixing approach (TSMA) in which all the aggregates are previously mixed dry, then half of the required water is added for a premixing stage and subsequently the cement is mixed followed by the addition of the remaining water and the completion of the mixing period (Fig. 18.2). The authors have reported that the premix stage leads to the formation of a thin layer of cement slurry on the surface of RA that permeates into the attached mortar filling up the old cracks and voids, which finally translates in concretes with stronger interfacial transition zones (ITZ) and higher mechanical and durability performance (Tam et al., 2005, 2007b; Tam and Tam, 2007). Tam and Tam (2008) also recommended the addition of silica fume and a percentage of the cement in the premix stage to further enhance the resulting ITZ and mechanical properties. Similarly, Kong et al. (2010) proved the effectiveness of the so-called triple method (TM), in which the wetted RA are superficially coated with pozzolans (fly ash, slag, or silica fume) prior to the addition of the cement and remaining water to the mix. Based upon the same principles, other arrangements of the TMSA and TM methods can be found in the literature (Li et al., 2009; Abd Elhakam et al., 2012; Wang et al., 2013; Mas et al., 2012).

Along the same lines of old mortar strengthening, a vast number of surface treatments, mainly coating alternatives, have been presented in the literature to minimize the adverse effects of the RA on the concrete manufacture. As seen in the mixing approaches (Tam and Tam, 2008; Kong et al., 2010), mineral additions such as fly ash, silica fume, etc. have been proposed as a cementitious surface improvement of RA since these materials are capable of strengthening the bond between old and new mortar and thus enhance the mechanic properties and microstructure of recycled concrete (Kou et al., 2011). To carry out the coating, Katz (2004) impregnated the dried RA in a silica solution (10:1 water-silica ratio and 1% superplasticizer) for 24 h and then the saturated RA was dried before using it again. Li et al. (2009) who compared fly ash, silica fume and blast furnace slag, also found this method to be an effective manner to hinder the water sorptivity of RA by creating a thin coating film made from pozzolanic powder. The authors stated that a dual coating with silica fume and fly ash



(i) NMA



(ii) TSMA

Fig. 18.2 Mixing procedures of the (i) normal mixing approach and (ii) two-stage mixing approach (Tam et al., 2005).

was the optimal approach and attributed the improved performance to their influence on the packing density achieved. Correspondingly, nanomaterials have also been exploited as a surface treatment to improve the RA performance. Zhang et al. (2016) who employed two different nanoslurries to coat the RA, observed a water absorption reduction of 43.75% for nanocalcium and nanosilica and 31.25% for nanosilica treatments. Similarly, Singh et al. (2018) reported that powdered nanosilica could be used to reduce the water uptake of RA up to 21.4%.

The use of different polymer-based impregnation products and methods has been tested by several researchers. Kou and Poon (2010) proposed RA to be soaked in poly-vinyl alcohol (PVA) solutions for 24 h inside desiccators with a vacuum pump (920 mbar). The authors found that 10% PVA was the optimum dosage to achieve a 44.33%–69.33% and 61.64%–74% water absorption reduction for 10- and 20-mm RA in oven-dry and air-dry conditions, respectively. Silicon-based polymers, which have water repellent properties, have also been studied (Kou et al., 2014; Zhang et al., 2015; Wang et al., 2016). Spaeth and Djerbi Tegguer (2013) compared a simple impregnation of 5 min soaking, drying at room temperature, 24 h, 20°C, 50% RH, and then in a 50°C ventilated oven, and a double impregnation method of 3 min soaking in soluble sodium silicate, drying at room temperature, 20 h, 20°C, 50% RH, followed by the steps in the simple method. Although the type of polymer solution and concentration affected the results, a clear water repellent behavior was observed for all treatments, and the single method showed the maximum reduction at 60% concentration, whereas the double treatment at 40% concentration led to water absorption values very close to that of NA. Santos et al. (2017), who soaked the RA for 24 h followed by 24 h drying at 105°C to accelerate the polymerization, found that a 20% silane emulsion resulted in a 45.52% and 64.62% WA reduction for concrete and ceramic RA, respectively. The authors, who also tested RA soaked (<10 min) in melted paraffin wax, reported water absorption reductions of 83.24% and 78.69% for concrete and ceramic RA, respectively. However, for both cases, the hydrophobic performance proved to be a transitory effect as it lost efficiency after a few minutes.

Carbonation of RA has also been suggested as a method to improve the properties of RA. Kou et al. (2014) designed a CO_2 curing method: vacuumed RA (-0.5 bar) were exposed to 100% CO_2 at 0.1 bar for 6–72 h (Fig. 18.3). For an optimum 24-h treatment, a water absorption reduction of 38%–54% and 38–48% for 10- and 20-mm RA, respectively, was seen. Following a similar approach, Xuan et al. (2017), who carried out the experiment at 0.1 and 5 bar, reported that higher carbonation pressures produced a marginal benefit to the process by around 1% water absorption reduction. For a simpler process in a carbonation chamber, 20°C, 60% RH at 20% concentration, a more modest response, between 23% and 28% water absorption reduction, was reported by Zhang et al. (2015).

Finally, a different technique arose in 2012, proposing the use of bacteria as a means to create a layer of calcium carbonate on the surface of RA through biodeposition in order to solve their water absorption drawback in the manufacture of concrete.

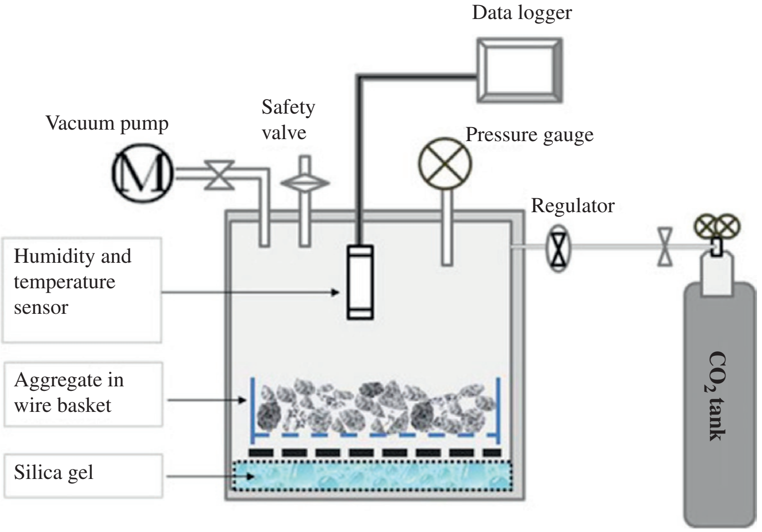


Fig. 18.3 CO_2 curing of RA (Kou et al., 2014).

18.3 Biodeposition on construction materials and bacteria-based self-healing concrete

Biogenic or microbial-induced calcium carbonate precipitation, sometimes known as BICP or MICP, has been proposed as an environmentally friendly technique to enhance the durability of stony materials due to its protective effect based on waterproofing and consolidating properties. According to Wang et al. (2016), this technique has been applied, basically in two different ways, initially as a consolidating surface treatment in façades, ornamental stones, statuaries, and other stony materials. Later other studies were developed, according to its potential, as an effective way to seal micro-cracks in cementitious materials, especially concrete and mortar, when the biological material is added in its mixture. The literature shows a number of works which have dealt with one, or both, techniques. An interesting example is the work published by Ramachandran et al. (2001), here the calcite bio-precipitation was applied using *B. pasteurii* in mortars as a surficial treatment and also adding the biomaterials in the mixing, both applications resulted in an improvement of durability.

Bacterially induced $CaCO_3$ precipitation has been proposed first for the protection of ornamental stones and limestone monuments. In situ applications were performed such as those studied (Castanier et al., 1999; Stocks-Fischer et al., 1999; Rodriguez-Navarro et al., 2003; Tiano et al., 1999), *inter alia*, which use this bio-technique to *medicate* monumental stones, façades, etc., leading to a further optimization and industrialization of the technique. Based on the collaboration between the French company Calcite Bioconcept, the Laboratory for Research of Historic Monuments and the University of Nantes (Le Métayer-Levrel et al., 1999), even a European patent (Adolphe et al., 1990) was developed using these types of biological treatment.

Subsequently, biodeposition was investigated as a surface treatment for cementitious materials (Ramachandran et al., 2001; De Muynck et al., 2008a; Ramakrishnan et al., 2005; Tiano et al., 1999; Dick et al., 2006). The general conclusions achieved in all these works are very similar, regardless of the species used (mostly *B. pasteurii* or *B. Sphaericus*). The general behavior of the material shows an important improvement in their water absorption, diminishing this characteristic values up to 68% less than control concrete; the volume of pores decreases, the mix achieves higher compressive strength and subsequently, durability acquires a better performance.

As explained above the starting of this practice was as an external or superficial treatment in stony materials. One of the first works (Le Métayer-Levrel et al., 1999) showed the potential to use carbonatogenic bacteria to precipitate calcite in natural stone, as a method of conservation or reparation. Many other subsequent studies have tried to demonstrate its efficiency as an external application in cementitious materials, as those reported in Bang et al. (2001), De Muynck et al. (2008a,b), Kim et al. (2013), Nosouhian et al. (2015), Ramakrishnan et al. (2005), and Van Tittelboom et al. (2010), most of these papers deal with conventional concrete or light concrete as study material, in others mortar is used as studied material. All these references have used different types of microorganism, especially bacteria of *Bacillus* or *Sporosarcina* genera; a more detailed study of the organisms used can be found in Section 18.4.

Some works were also focused on how to improve the method of application, immobilizing the bacterial strains in certain media, which can facilitate their use. For example, the work shown in Bang et al. (2001) used polyurethane foam to improve $CaCO_3$ deposition. Nevertheless, in order to improve the crack repair technique some other researches (Van Tittelboom et al., 2010) have used silica gel as a media to fix and keep in good conditions the bacterial strains until they start their activity (a more detailed explanation is offered in the next paragraphs). The general conclusions achieved in these chapters are that the use of superficial application of carbonatogenic bacteria could present a similar behavior as other external conventional treatments, but avoiding the negative side-effects, using a natural and environmental compatible method; obtaining an important reduction of the water absorption of the materials, diminishing chloride migration, increasing the resistance to freeze-thaw cycles and improving durability in alkaline or acid environments.

As previously commented, another series of works (Achal et al., 2011; Ghosh et al., 2005, 2009; Luo et al., 2015; Wang et al., 2012, 2014) have focused the research on how to develop self-healing materials. This capacity implies the possibility of the material to acquire an endogenous skill of healing itself through the use of calcite or silica biodeposition. In such a way, similar bacterial strains have been used. To reach this skill it is necessary to add the microbial spores into the mixing mass, so this capacity remains latent until a crack or fissure appears, in that moment the increasing of moisture can activate the latent strains, which will start to bio-produce the calcium carbonate. All the previously cited studies have added the microorganism strains during the mixing process of mortar or concrete in a plastic state, so that the incorporation of the biogenic material is relatively easy to obtain at the end of the mixing stage a “microbial” mortar or concrete (Achal et al., 2011). The majority of the studies have used some *Bacillus* sp. as bio-healing agent, but some other microbial strains of *Shewanella* genre and even *Escherichia coli* (Ghosh et al., 2005, 2009) have also been used, in this case, the *Shewanella* sp. have the ability to bio-deposit silicates. Also noticeable are the findings achieved by Ghosh et al. (2009), who have tried to study the protein biological mechanisms of biodeposition, in order to get a possible molecular isolation of these types of protein. Thus, it would be possible to add the carbonatogenic protein instead of the biological agent, so that the process could be simpler. Although this study may open a future trend for self-healing materials, this possibility is still not developed in-depth. Nowadays it seems that *Bacillus sphaericus* specie is the most suitable one, due to its high capacity to deposit $CaCO_3$. In a similar way to the superficial application for cementitious materials, the majority of these studies have been developed in concrete and a few in mortar.

Indeed this technique can develop two different effects: the self-healing ability is produced, as explained above when microbial agents added in the mixture (and sometimes immobilized in a media like polyurethane foam or silica gel) are put in contact with external moisture when a superficial crack appears in the material. Subsequently, when the biodeposition starts the calcium carbonate heals or close cracks between 0.5 and 0.8 mm width according to some studies. The calcite biodeposition develops an important decrease of the capillary water absorption of the concrete or mortar samples and the biodeposition seals part of the cementitious matrix pores, obtaining a more impermeable material. The second effect when a microbial concrete is made, is that the mechanical strength of the material increases, this is also due to the bio-precipitation of calcite or silica in the matrix, which, according to some studies (Achal et al., 2011), can provoke a 36% higher compression strength in the test samples.

Several works have focused on the biological mechanisms used by ureolytic bacteria, since these kind of bacteria are able to induce the precipitation of calcium carbonate by the production of the urease enzyme, which catalyzes the hydrolysis of urea to CO_2 and ammonia, resulting in an increase of the carbonate concentration and the pH in the bacterial environment (Castanier et al., 1999; Hamilton, 2003; Stocks-Fischer et al., 1999; Siddique and Chahal, 2011). Most of these biochemical and biological mechanisms have proven their efficiency to obtain carbonate or bicarbonate ions and precipitate solid particles of these minerals by heterotrophic bacteria. The metabolic pathways are mostly based on the nitrogen cycle. As explained in some studies (Castanier et al., 1999; Hamilton, 2003) carbonatogenesis is neither restricted to a particular taxonomic group of bacteria nor to specific environments, so is a ubiquitous phenomenon produced in the Earth, in a natural way since the Precambrian. Another important factor is an in-depth study of the ionic interchange between the cell membrane and the external environment, despite the general mechanism is well known, and a number of studies describe it with detail, the cellular changes are still poorly known. Many bacteria strains have demonstrated their natural ability to produce calcite biodeposition, and even according to Siddique and Chahal (2011) it has been hypothesized that almost all bacteria are capable of $CaCO_3$ production because precipitation occurs as a byproduct of common metabolic processes such as photosynthesis, sulfate reduction, and urea hydrolysis. Despite this effect is very common in nature, some studies (Stocks-Fischer et al., 1999; Siddique and Chahal, 2011) have focused on finding the best microbial strain to obtain a real healing effect in stony materials such as natural stone, mortar, concrete, etc., finding that *Bacillus* sp., and especially *B. pasteurii* and *B. sphaericus*, are the more effective and adequate species.

Maybe the most used bacillus strain used for these studies has been *Bacillus sphaericus*. Its urease activity and also its capacity to precipitate calcium carbonate have made that this a preferred specie to develop in number of studies (De Muynck et al., 2008a,b; Kim et al., 2013). In one of them, *Bacillus sphaericus* was used as superficial bio-treatment in a mortar, normal concrete and lightweight concrete, comparing its efficiency as external layer protector with some conventional agents. The results obtained in these works showed that protection obtained through biodeposition of calcium carbonate acts efficiently in a similar way to conventional agents, avoiding some negative environmental effects and even reducing the water absorption of hardened concrete up to 90% compared with nonbiotreated concrete.

Other studies have used *Bacillus sphaericus* as superficial bioagent to protect and consolidate ornamental limestone (Rodriguez-Navarro et al., 2003; Dick et al., 2006). This microorganism has shown higher efficiency than other Bacillus strains, for example, *B. lentus,* and can produce a protective layer in façades, historical buildings and, in general, in some degraded limestone pieces. Rodriguez-Navarro et al. (2003) compared the bio-formation of calcium carbonate between some Bacillus strains and another microorganism *Myxococcus xanthus*, a Gram-negative myxobacteria. This organism is able to produce an efficient bio-cementation in ornamental stone through deposition of $CaCO_3$, getting better consolidation of degraded stone layers than *B. sphaericus* and without plugging the stone natural pores. Besides, when the microorganism ends its biodeposition, the strain dies and so it is possible to avoid an uncontrolled bacterial growth, this fact can appear with the use of other bacteria, as *Bacillus* sp.

Finally, other studies have focused on the self-healing properties of *Bacillus sphaericus* when added to concrete mixtures. In the work of Wang et al. (2014), an effective self-curing treatment is applied by hydrogel particles containing *B. sphaericus*. The hydrogel acts as a water reserve to get autogenous and microbially induced healing actions through the deposition of calcium carbonate in hardened concrete. This method allowed sealing 0.5-mm wide cracks in the concrete samples.

18.4 Biodeposition treatment on recycled aggregates

The *Bacillus* group has been considered as one of the most suitable kinds of microorganism to reach a biologically induced mechanism of calcium carbonate precipitation (De Muynck et al., 2010; Wong, 2015), since they are found amply in natural environments, they can be easily cultivated and they show a noteworthy potential to create large volumes of calcite in a relatively short time. The mineral produced is highly dependent on the environmental conditions (Rivadeneyra et al., 1994) and no specialized cell structure or specific molecular mechanism is thought to be involved (Barabesi et al., 2007).

The promising results of this technique have encouraged some research groups to evaluate the effect of $CaCO_3$ biodeposition on recycled coarse aggregates to be used in recycled concrete. This technique could solve the problem of high water absorption values associated with this kind of aggregates (Grabiec et al., 2012; Qiu et al., 2014; García-González et al., 2017; Wang et al., 2017).

In the experiments developed by Dick et al. (2006) on degraded limestone, *B. sphaericus* was used to produce calcium carbonate. According to this study, this microorganism is able to precipitate calcium carbonate $CaCO_3$ on its cell constituents and in its microenvironment by decomposition of urea $CO(NH_2)_2$ into carbonate CO_3^{2-} and ammonium NH_4^+. Subsequently, the carbonate promotes the microbial deposition of $CaCO_3$ in an environment with the high calcium content.

According to the methodology explained on the paper of García-González et al. (2017), to grow *B. sphaericus*, the liquid culture media used consisted of autoclaved yeast extract and filter sterilized urea solution. The final concentrations of yeast extract and urea were 20 g/L. The pH of the medium was 7. Cultures were obtained after two times successive culturing and 1% inoculums from a −80°C stock culture. Then they were incubated at 28°C on a shaker at 100 rpm for 24 h. The concentration of the bacteria cells in the *B. sphaericus* culture was around 10^8 cells/mL and a pH value of around 9.3.

Biodeposition treatment of the recycled aggregates was performed in an air-conditioned room (20°C, 60% RH) under static and nonsterile conditions, that is to say, open to the air. Firstly, dry recycled aggregates were immersed in one-day-old *B. sphaericus* grown culture for 24 h of incubation period. Subsequently, aggregates were removed from the culture solution. Secondly, aggregates were immersed for 4 days in a deposition medium containing 12 g/L of urea, 47 g/L of $Ca(NO_3)_2.4H_2O$ and 5 g/L of yeast extract. After this immersion time, the aggregates were gently washed and drained to remove calcium carbonate precipitate which had a weaker bond to the surface of the aggregate.

18.5 Effects of bio-based self-healing agents on recycled aggregates

Two mixed recycled aggregates (RA1 and RA2 samples) and one ceramic recycled aggregate (RA3 sample) were biotreated. Their properties and composition are summarized in Tables 18.1 and 18.2.

The characterization of the recycled aggregates after biodeposition treatment is discussed below.

18.5.1 SEM characterization

By SEM analysis, it is observed how the calcium carbonate deposition over the surface of the aggregate is a layer with a uniform and low thickness (Fig. 18.4). Similar results were published by Kim et al. (2013), who used biodeposition of *B. sphaericus* and *B. pasteurii* to treat normal and lightweight concrete samples. The present study displays substantially more regular $CaCO_3$ crystals (Fig. 18.5).

Table 18.1 Properties of RA samples.

	RA1	RA2	RA3
Maximum particle size (mm)	16.00	16.00	16.00
Minimum particle size (mm)	4.00	4.00	4.00
Granulometric modulus	7.67	7.75	7.96
Fines content (%)	0.04	0.87	0.72
Apparent density (Mg/m^3)	2.53	2.51	2.65
Oven-dry density (Mg/m^3)	2.08	2.06	2.29
Saturated surface dry density (Mg/m^3)	2.26	2.24	2.43
Flakiness index (%)	14.75	19.10	24.67
Los Angeles coefficient (%)	40.99	39.00	33.23

Table 18.2 Composition of RA samples.

Component (%)	RA1	RA2	RA3
Ru: Unbound natural aggregates	17.5	22.5	0.0
Rb: Ceramic materials	33.6	38.4	97.9
Rc: Concrete and mortar	44.1	37.1	2.1
Ra: Asphalt	0.4	1.7	0.0
Rg: Glass	0.8	0.0	0.0
X: Others (gypsum)	3.6	0.3	0.0

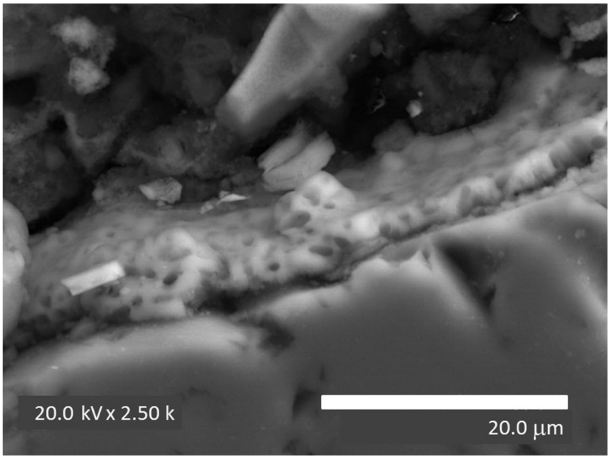


Fig. 18.4 $CaCO_3$ precipitate over the aggregate surface.

The calcium carbonate stratum looks like a white compact agglomeration of globular structures (Fig. 18.5), it is for that characteristic appearance that the microbial biodeposition of *B. sphaericus* is easy to recognize on the recycled aggregates. The globular structures show the imprints of bacterial cells involved in $CaCO_3$ precipitation, being more or less widespread according to the bacterial activity in each area (Fig. 18.5).

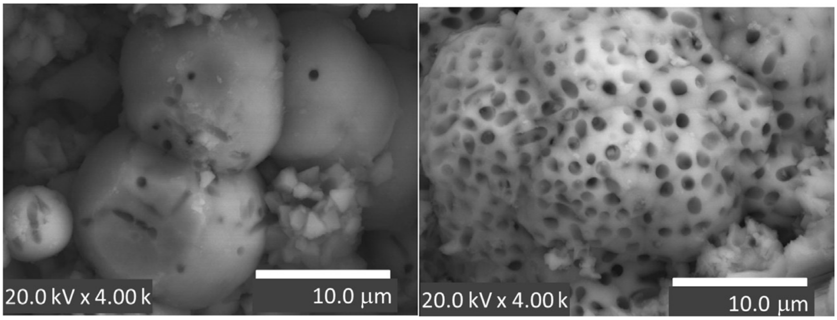


Fig. 18.5 $CaCO_3$ precipitate with different bacterial activity.

Spherical crystals due to the biodeposition of *B. pasteurii* were shown in the study of Qiu et al. (2014), however, the crystals had a smooth surface without the empty spaces that were noticed in the current *B. sphaericus* deposition. In addition, the spherical structures from *B. pasteurii* were distributed with less compactness than the crystals from *B. sphaericus* seen in this study. The presence of crystals of calcium carbonate from *B. pasteurii* over the surface of recycled concrete aggregates was also referred by Grabiec et al. (2012), but in their case, the $CaCO_3$ crystals displayed more irregular shapes than the deposition evaluated in the present research. This variability between different papers may be explained by the biotreatment conditions and the nutrients used during the cultivation since authors like De Muynck et al. (2008a) confirmed that the type of medium composition had a profound influence on the morphology of calcium carbonate crystals.

Fig. 18.6 makes it possible to see the partial pore-filling effect of the biodeposition treatment on recycled aggregates, since globular calcium carbonate crystals have been detected on the internal pore surface of a recycled ceramic aggregate decreasing the permeability of the biotreated aggregate. When the calcium carbonate layer over the aggregate surface and the pore-filling effect are taken into account, a waterproofing

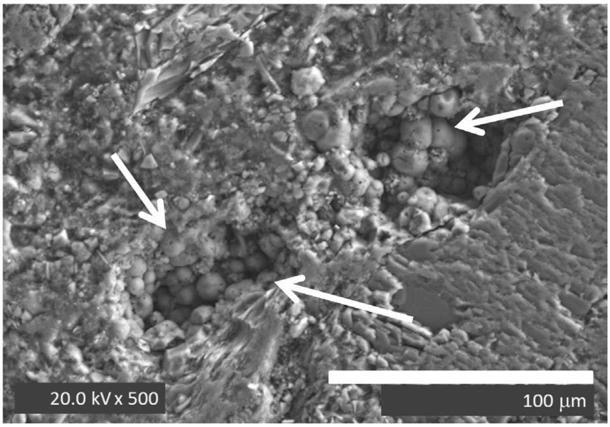


Fig. 18.6 Aggregate pores filled by $CaCO_3$ precipitate.

result is expected, which will be concordant with the reduction of the water absorption values shown below for biotreated aggregates.

EDX results (Fig. 18.7) show the composition of the globular shapes by EDX analysis, the main component being calcium, followed by oxygen and carbon. These results confirm that they are likely to be $CaCO_3$ precipitate.

18.5.2 Weight variation

To test the weight change, two subsamples were taken from each RA studied sample (one subsample with a particle size of 4–12.5 mm, further referred to as S group, and the other sample with a particle size 12.5–20 mm, called L group). The biodeposition treatment increased the weight of all RA tested samples (Fig. 18.8). This weight gain depends on several factors. First, the greater percentage of weight increase was detected in the samples from the S group, 16%–46% higher than for the L group. This may be caused by the higher surface/volume ratio of the S group samples, in comparison with the samples of the L group, and taking into account that biodeposition is considered as a surface treatment by several authors (Qiu et al., 2014; De Muynck et al.,

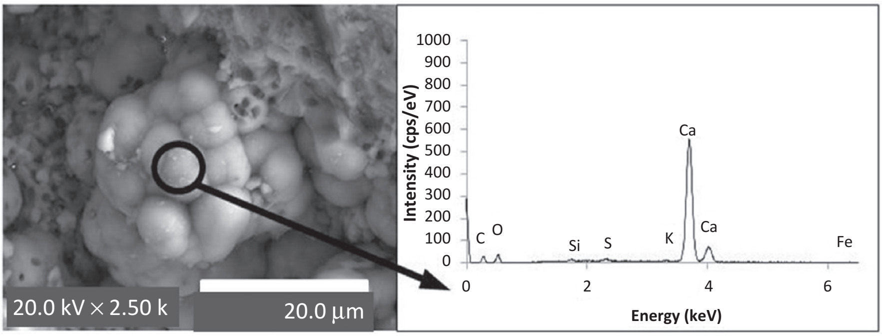


Fig. 18.7 SEM image and EDX spectra of $CaCO_3$ precipitate on biotreated RA.

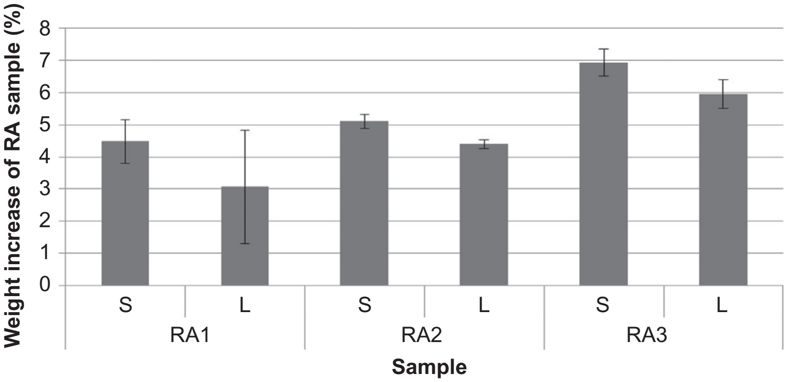


Fig. 18.8 Weight increase of RA samples after biotreatment.

2008a, 2013). This statement was confirmed in the present paper by SEM analysis (see above). Secondly, the weight increase was growing high with the rise of ceramic content in the RA sample. This is probably related to the roughness of the RA particles, considering that the ceramic aggregate surface has a greater roughness than unbound natural aggregates. Therefore, the biodeposition is easier on the surface of ceramic and cement-based particles. In addition, ceramic aggregates roughness is lower than that shown by old concrete particles; this fact has been qualitatively checked by SEM analysis, as shown in Fig. 18.9. The surface of these concrete particles seems too irregular to allow the deposition of calcium carbonate in larger $CaCO_3$ layers, whereas the less irregular ceramic surface allows the generation of a more continuous calcium carbonate layer. These results are in concordance with the information obtained from the sonication test of the current research.

All test samples have shown higher weight gain than the material tested in the published research of Qiu et al. (2014). Results of this test displayed a weight increase of 1.03% in the 100% concrete recycled aggregates of 5–20 mm particle size, when microbial carbonate precipitation was carried out with bacteria concentration of 10^8 cells/mL, at 35°C and pH 9.5 and with a solution containing 16.8 g/L of calcium. Their results with lower values of weight increase may be related to the lack of ceramic particles in their samples.

It is important to highlight the observation of some authors (Chunxiang et al., 2009; Okwadha and Li, 2010; Otlewska and Gutarowska, 2016), who state the significant effect of calcium concentration and method of calcium and urea addition on the biodeposition treatment results. Previous studies of the authors of this chapter (Wang et al., 2012, 2014; De Belie and De Muynck, 2010; Zachar et al., 2010) have reported that the chosen parameters for biodeposition treatment in the current research are suitable to achieve an optimal microbially induced carbonate precipitation for concrete applications.

18.5.3 Water absorption

The results of water absorption of the RA before and after biodeposition (Fig. 18.10) show that this technique reduces the water permeability of all treated samples. Although this happens only to a limited extent going from 3 % decrease for S samples of RA2 to 10% for L samples of RA3 and 18% for L samples of RA1.

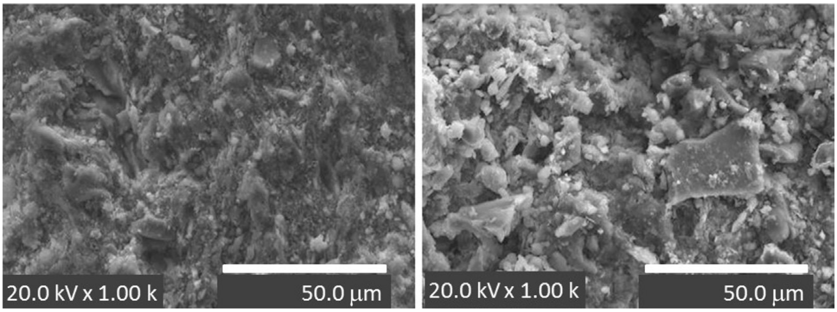


Fig. 18.9 Surface SEM images of ceramic (left) and concrete (right) recycled aggregates.

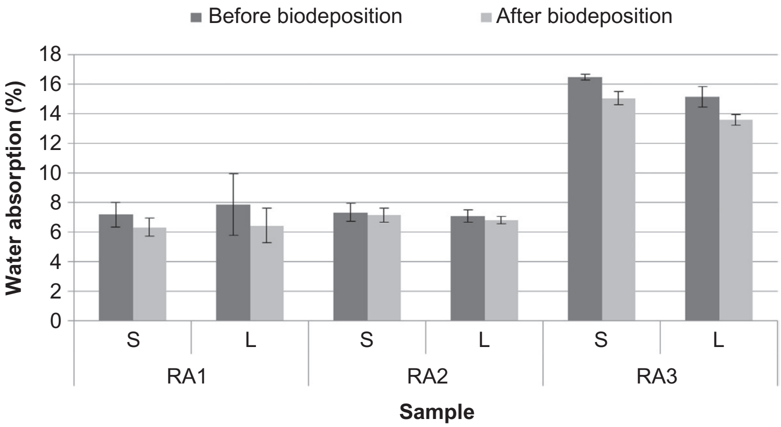


Fig. 18.10 Water absorption of recycled aggregates before and after biotreatment.

The water absorption reduction may also be dependent on the composition of recycled aggregates. The test results pointed to a lower water absorption reduction when the unbound natural aggregates content in the samples was greater: RA2 sample with the highest content of unbound natural aggregates according to Table 18.2 shows the lower reduction, 2.7% for the S group and 3.8% for the L group. However, samples with a higher content of cement-based particles, RA1 sample, showed a greater reduction of water absorption: 11.7% for the S group and 17.9% for the L group.

This behavior could be caused by the roughness and the porosity, especially macroporosity, of the different constituents present in the recycled aggregates since the roughness and the porosity of cement-based particles is higher than that of unbound natural aggregates (De Brito et al., 2005; Limbachiya et al., 2000; Poon et al., 2004a; Sagoe-Crentsil et al., 2001; Evangelista and de Brito, 2007). According to the research of Declet et al. (2016), the surface roughness can affect the adhesion of the calcium carbonate precipitate onto the aggregate surface, therefore it is expected to be more powerful on ceramic and cement-based particles. Also, the higher porosity and roughness of ceramic and cement-based particles than these properties in the unbound natural aggregates affect the creation of $CaCO_3$ precipitate because more calcium carbonate can be deposited on the more rough and porous surfaces, where the less irregular surface of ceramic particles allows the generation of a more continuous calcium carbonate layer. The greater macroporosity of cement-based particles in comparison with ceramic ones, as shown in the MIP analysis (Fig. 18.11), justifies that samples with more concrete and mortar particles show the higher waterproofing effect since greater $CaCO_3$ is deposited inside their superficial macropores. On the surface of ceramic particles a more continuous biofilm is formed, nevertheless this kind of material offers a lower pore-filling effect.

Grabiec et al. (2012) and Qiu et al. (2014), who used the microorganism *B. pasteurii*, stated that the biodeposition process led to a reduction in the water absorption of recycled aggregate produced by crushing 100% old concrete, testing different bacteria concentrations and recycled aggregate qualities. Gabriec et al. detected a water

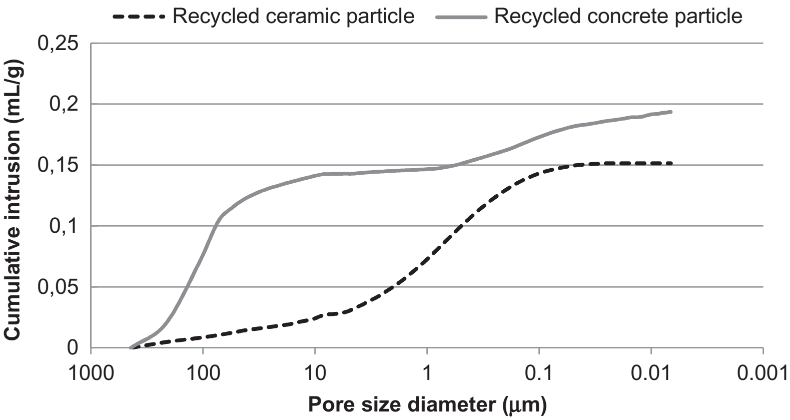


Fig. 18.11 Pore size distribution of ceramic and concrete recycled particles.

absorption decrease of 30%–35% for aggregates of particle size 6/8 mm, and 50% drop in water absorption for particle size 12/16 mm when a bacteria concentration of 10^7–10^8 cells/mL was applied at 30°C room temperature. Qiu et al. evaluated the effect of biodeposition on recycled concrete aggregates of particle size 5–20 mm, showing that a treatment with bacteria concentration of 10^6 cells/mL, at 25°C, pH 8.2 and 5.6 g/L of calcium content, decreased the water absorption of recycled aggregates with 8%. When the bacteria concentration was 10^8 cells/mL, and application conditions were 35°C, pH 9.5 and 16.8 g/L of calcium content, the water absorption was reduced by 8%–16%. The great effect of water absorption decrease published by these authors could be justified because they were testing 100% concrete recycled aggregates.

Porosity testing of ceramic and cement-based particles from recycled aggregates supports the stronger pore-filling effect by calcium precipitate on recycled concrete aggregates, and as can be seen in Fig. 18.11, particles from old concrete show greater porosity, with especially a greater amount of pores with a diameter larger than 1 µm. On the other hand, the pore size distribution of ceramic particles from RA displays a lower presence of macropores, which results in a limited deposition of calcium carbonate inside the pores, although a more continuous carbonate layer is formed and the adhesion to the particle surface of intermediate roughness is better than for recycled concrete aggregates.

18.5.4 Resistance to sonication

The resistance values against ultrasonic attack achieved by the RA samples (Fig. 18.12) show that the sample with higher content of cement-based particles (RA1) displays 44 % less weight loss upon sonication of treated aggregates than untreated aggregates in case of L size, and 6% for the S size. This can be compared with the findings of De Muynck et al. (2011), who support that the concrete prisms treated with bacteria in

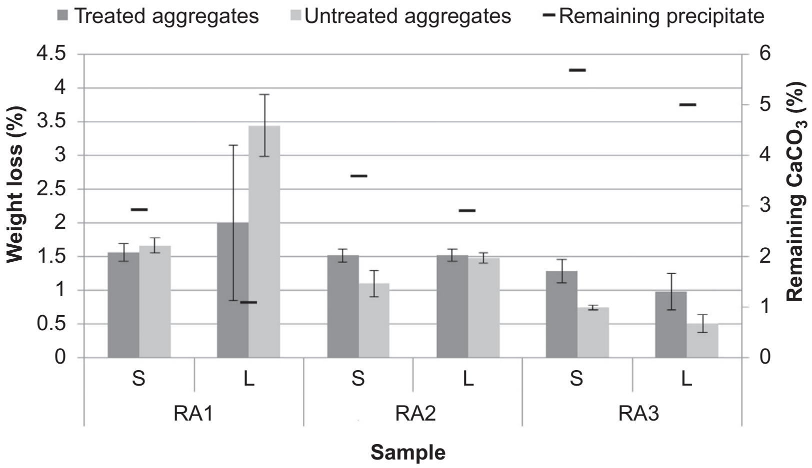


Fig. 18.12 Weight loss of treated and untreated RA samples after six cycles of sonication, and the remaining amount of $CaCO_3$ of treated samples.

culture liquid and biodeposition medium exhibited greater resistance to sonication than untreated prisms. The consolidating effect can be associated with the stronger compaction level that calcium precipitate provides to the cementing layer on the surfaces of the concrete or mortar aggregate after cracks filling. In the same way as for the water absorption results, this positive effect is more obvious for the larger particles because they have wider and longer cracks and a higher quantity of calcium carbonate can be deposited on those surface cracks.

When the ceramic content increases in the sample (RA3), the resistance to sonication increases for both treated and untreated aggregates. For these RA the biotreated aggregates exhibit greater weight loss than untreated aggregates, 74% higher in case of S size and 98 % for the L size. However, since more $CaCO_3$ has been deposited on the ceramic aggregates in the first place (Fig. 18.8), the remaining amount of carbonate deposition would be still much larger than for the RA with a lower ceramic fraction (Fig. 18.12).

18.6 Effects of bio-based self-healing agents on recycled concrete

Using the RA1 sample, recycled mixed aggregate, these aggregates were biotreated and added to the concrete mix to evaluate their effect on the manufactured material. Then, two concrete samples were tested in this section, one control concrete with recycled aggregates in their original state, control recycled concrete CRC, and a second concrete manufactured with biotreated recycled aggregates BRC. The results are discussed below.

18.6.1 Fresh concrete characterization

18.6.1.1 Consistency

The use of biodeposition on recycled aggregate before the addition to the concrete mix is able to improve its workability noticeably by 139%. Using concrete with recycled mixed aggregate without biodeposition treatment, the control sample develops a dry level of consistency with slump S1. However, the concrete with biotreated aggregates achieves a slump of plastic level S2, according to the standard EN 12350-6 (2009).

18.6.1.2 Fresh density

The presence of calcium carbonate deposition is almost imperceptible on the possible variation of the fresh concrete density evaluated according to the standard EN 12350-6 (2009), being 0.3% lower in case of concrete with biotreated aggregates in comparison with the control sample.

18.6.2 Chemical and microstructural characterization of hardened recycled concrete

SEM analysis allows observing the $CaCO_3$ distribution over the aggregate surface, both recycled and natural (Fig. 18.13), and inside their pores, confirming the evidence that biodeposition technique is a suitable method to decrease the water absorption of the aggregates since it is able to precipitate calcium carbonate and fill in interior pores. Kim et al. (2013) and Qiu et al. (2014) also claimed this impermeability effect.

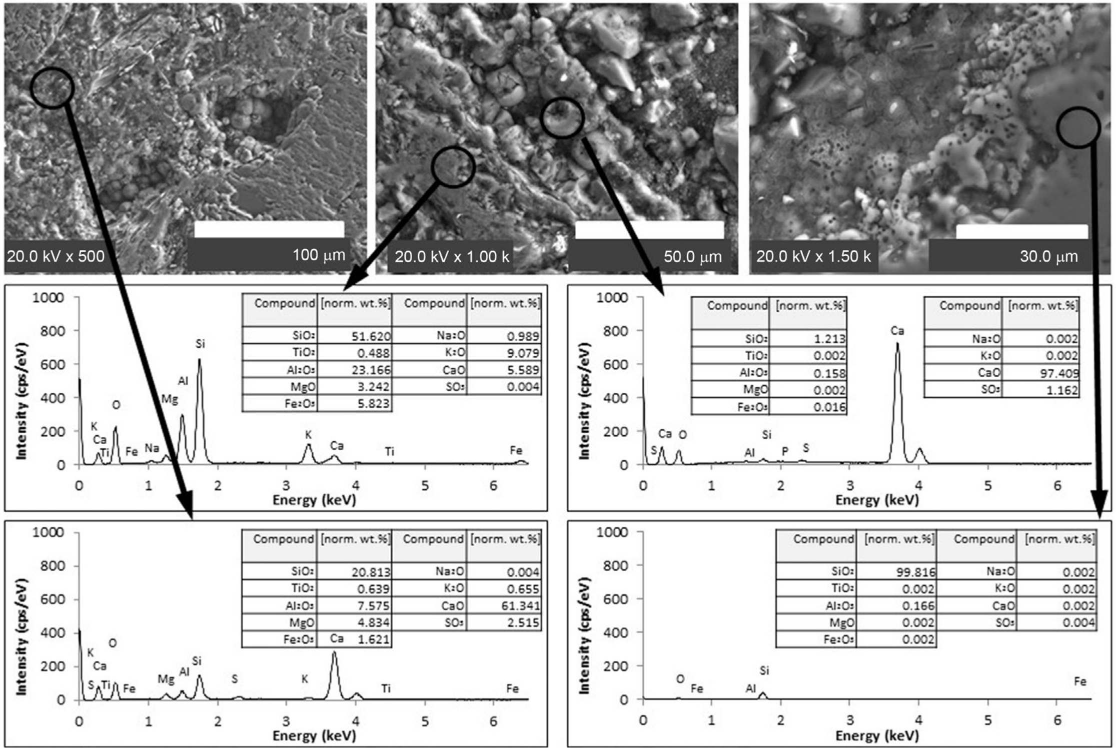
EDX spectrums of BRC samples (Fig. 18.13) show the presence of calcium carbonate precipitation by Ca picks, increasing the Ca/Si ratio of the concrete blend.

Element maps of BRC sample at 90 days display the physical connection between the calcium carbonate and the recycled aggregate in Fig. 18.14, which is better than that between $CaCO_3$ precipitate and the natural aggregate, observed in Fig. 18.15. Ca element distribution (pink color) has a stronger contrast when it is precipitated around natural aggregate (Fig. 18.15). However, pink color mixed up with recycled aggregate surface and its cracks are shown in Fig. 18.14, where a recycled aggregate is mapped.

18.6.3 Mechanical characterization of hardened recycled concrete

18.6.3.1 Compressive strength

Fig. 18.16 shows the values of compressive strength developed by the CRC and BRC samples at 7 and 28 days according to the standard (EN 12390-3, 2009), where can be seen that the sample reaches a compressive strength 15% higher than the CRC sample at 7 days, however, at 28 days this behavior is inverted and CRC sample displays a greater strength level, 22% higher than BRC sample. In any case, both types of concrete exceed by 6.8% the 25 MPa of target strength at 28 days for recycled concrete. This behavior could be explained by the lower water absorption of the biotreated recycled aggregate,



Compound	[norm. wt.%]	Compound	[norm. wt.%]
SiO_2	51.620	Na_2O	0.989
TiO_2	0.488	K_2O	9.079
Al_2O_3	23.166	CaO	5.589
MgO	3.242	SO_3	0.004
Fe_2O_3	5.823		

Compound	[norm. wt.%]
SiO_2	1.213
TiO_2	0.002
Al_2O_3	0.158
MgO	0.002
Fe_2O_3	0.016

Compound	[norm. wt.%]
Na_2O	0.002
K_2O	0.002
CaO	97.409
SO_3	1.162

Compound	[norm. wt.%]	Compound	[norm. wt.%]
SiO_2	20.813	Na_2O	0.004
TiO_2	0.639	K_2O	0.655
Al_2O_3	7.575	CaO	61.341
MgO	4.834	SO_3	2.515
Fe_2O_3	1.621		

Compound	[norm. wt.%]	Compound	[norm. wt.%]
SiO_2	99.816	Na_2O	0.002
TiO_2	0.002	K_2O	0.002
Al_2O_3	0.166	CaO	0.002
MgO	0.002	SO_3	0.004
Fe_2O_3	0.002		

Fig. 18.13 SEM images (magnification of 500×, 1000×, and 1500×) and EDX spectrums of BRC sample at 90 days.

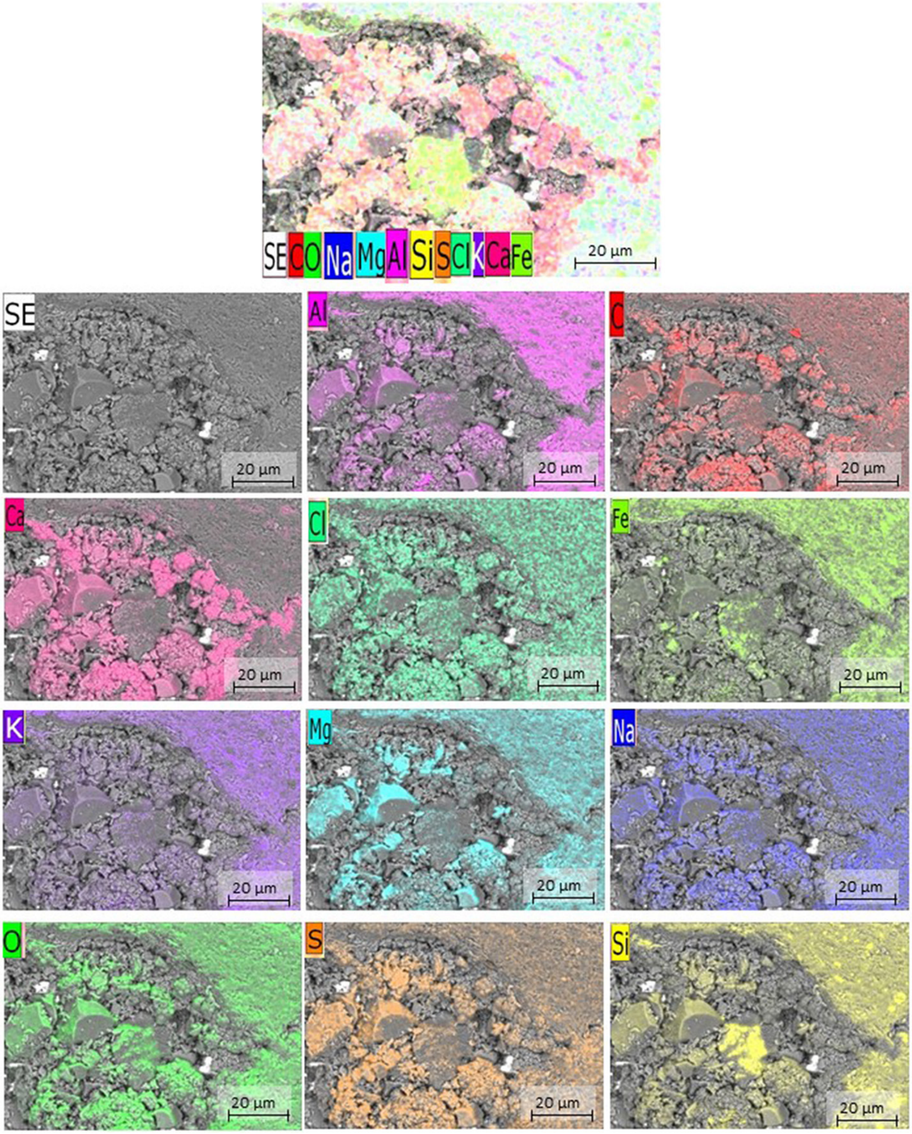


Fig. 18.14 Element maps of BRC sample at 90 days (magnification of 350×).

so that greater water quantity could be available in the concrete paste to accelerate the formation of cement hydration products, increasing the compressive strength of the concrete sample at early age. Over time, the lower effective W/C ratio in the paste of CRC sample causes a higher compressive strength level than BRC sample. Water corrections for obtaining the same slump could improve the BRC strength over time.

Jonkers and Schlangen (2008) found an adverse effect of bacteria or organic substances presence on mortar mixtures when their compressive strength was evaluated

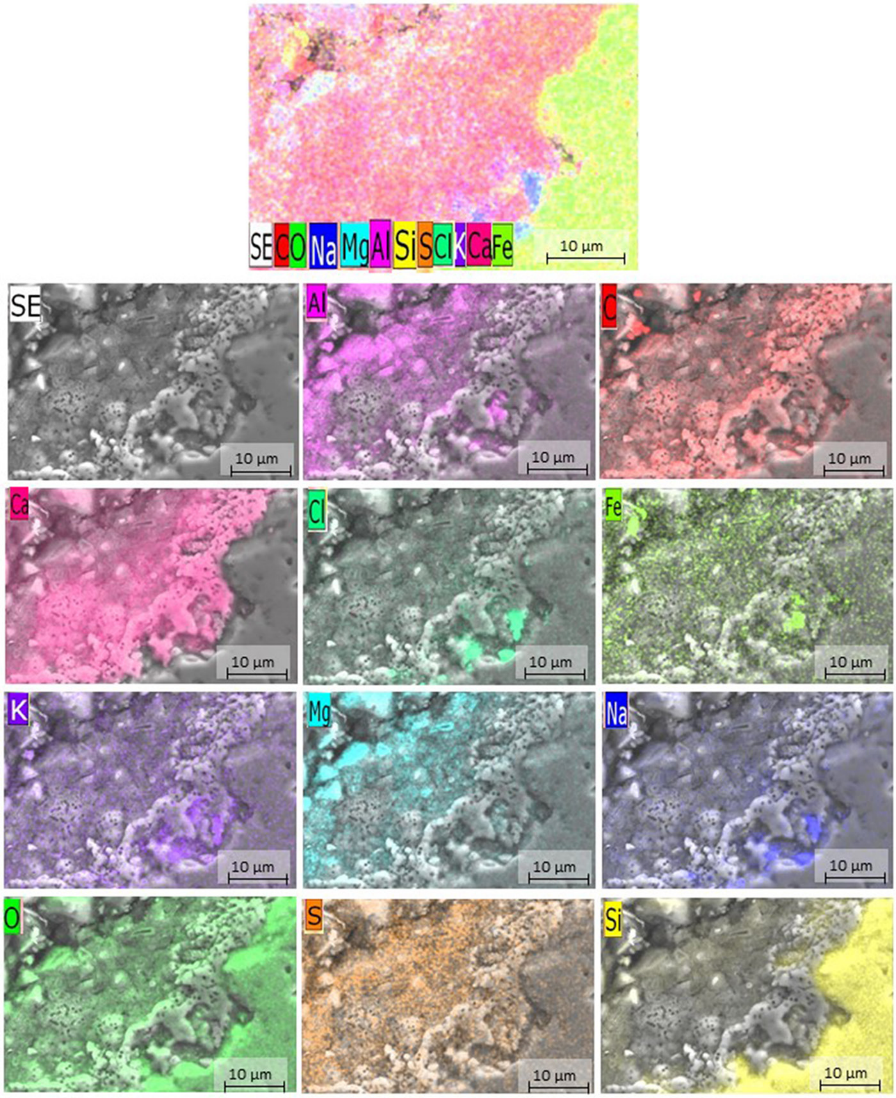


Fig. 18.15 Element maps of BRC sample at 90 days (magnification of 1500×).

at 28 days of material curing. However, Wang et al. (2017), who made concrete with recycled mixed aggregates and recycled concrete aggregates, both treated by a similar biodeposition technique than the used one in this study, claimed that compressive strength of manufactured concrete was increased by 40% when concrete aggregates were added and 16% when mixed aggregates were used (Fig. 18.17).

Taking into account of these findings, the compressive strength decrease obtained by the concrete sample with biotreated aggregates in this study could be improved by adjusting the W/C ratio of the concrete mix.

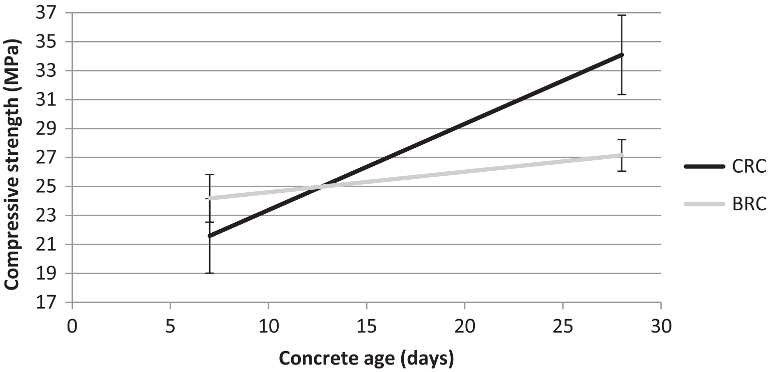


Fig. 18.16 Compressive strength of CRC and BRC samples at 7 and 28 days.

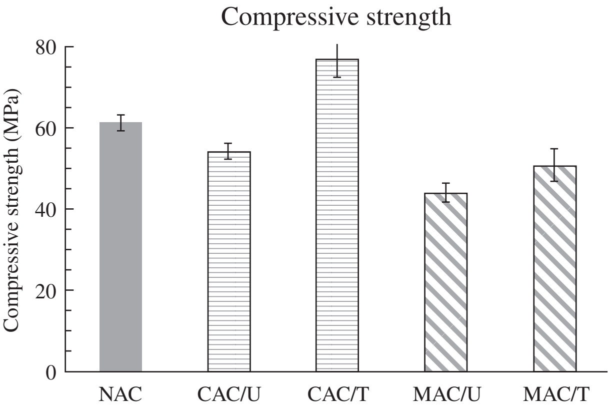


Fig. 18.17 Properties of concrete made with natural aggregates (NAC), untreated recycled concrete aggregates (CAC/U), treated recycled concrete aggregates (CAC/T), untreated recycled mixed aggregates (MAC/U), and treated recycled mixed aggregates (MAC/T) (Wang et al., 2017).

18.6.3.2 Density of hardened concrete

Following the same behavior than the fresh concrete density, the value of the hardened concrete density at 28 days for the biotreated sample is slightly lower, 3.2%, than the control one, both tested according to the standard EN 12390-7 (2009). This could be due to a higher effective W/C ratio.

18.7 Future trends

The development of new construction and building materials has gone through a big evolution in the last decades: more resistant and durable products, with higher capacities and increased versatility have been developed. Construction techniques and the civil engineering discipline as a whole reached a level that was unthinkable some years ago and has touched with other domains, leading to multidisciplinary solutions to material and structure related problems. Within these new trends, one of the most novel has been the emergence of self-healing materials, which can present an endogenous capacity of crack healing, avoiding expensive maintenance operations during the service life of the construction. To reach this ability, biological agents have been used, in the so-called biomaterials. Mostly, but not only, these agents are bacteria, whose enzymatic capacities have been taken advantage of to get biological deposition of calcium carbonate, protecting stony materials. Other species have also been used, as fungi and diatom algae, the latter able to produce silica through its frustules. All these bio-techniques constitute a promising scenario for the development of new self-healing materials, and besides allow a wide range of possibilities in their application. The use of biodeposition for consolidation of recycled aggregates is an option to consider here. With the aim to increase the sustainability of the construction industry, the use of recycled materials as aggregates has a high potential. However, due to the high water absorption compared with traditional natural aggregates, the recycled aggregates generally lead to a decrease in concrete properties. Hence, recycled aggregates benefit from a bacterial treatment, improving their density and the properties of the concrete in which they are introduced.

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