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Modification of recycled concrete aggregate by calcium carbonate biodeposition

Anna M. Grabiec [a,*], Justyna Klama [b], Daniel Zawal [a], Daria Krupa [a]

[a] Department of Theory of Structures and Agricultural Building Engineering, Faculty of Land Reclamation and Environmental Engineering, University of Life Sciences, Poznan, Poland
[b] Department of General and Environmental Microbiology, Faculty of Agronomy and Bioengineering, University of Life Sciences, Poznan, Poland

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ABSTRACT

A growing demand for raw materials leads to danger of premature depletion of the natural sources. An alternative is to use by-products, provided their quality is improved. The paper presents surface modification of recycled aggregate concrete using biodeposition involving a method employing *Sporosarcina pasteurii* (*Bacillus pasteurii*) bacteria. It was possible to obtain reduction in water absorption of aggregate, the effect was more visible in case of finer fractions and for aggregates originating from inferior quality concrete. Calcium chloride was used for precipitation of calcium carbonate, while culture medium consisting of beef extract, peptone and urea was used for cultivation of microorganisms. In addition, whey, ecologically dangerous by-product from dairy industry was found to be effective as a culture medium. Presence of calcium carbonate crystals covering aggregate grains was confirmed by observations under scanning electron microscope. In the perspective, the proposed method, upon appropriate improvements, seems worthwhile due to ecological and technological reasons.

1. Introduction

Development achievements, both in civil engineering or any other area still remain out of reach for the great percentage of general population, while on the other hand technologies employed are causing havoc on the environment that should serve the greatest good of humanity. For more than a decade, every year worldwide, over 1 m^3 of concrete per capita is produced [1]. This requires the use beyond 2 billion tons of cement, which means some 7% of global carbon dioxide emissions, estimated in total to be approximately 30 billion tons.

The use of concrete aggregate for concrete production also constitutes a significant factor that poses a threat to the environment. Demand for this material has to be met by exploring newer and newer fields, and this leads to rising economic costs and frequent protests from a society that is becoming more and more informed. For example, in Poland a plan to establish one of the new sand pits in the Zabnik Valley sanctuary forest was effectively blocked by the local population who pointed out the threat posed to the survival of specific species of birds in this region (www.jaw.pl/video/show/11296/). Aggregate might be obtained also from the seas and oceans. That, however, poses a problem due to the presence of chloride ions, which must not be used to produce reinforced concrete. In the extreme cases this could cause building disasters. Therefore, interrelations in human life environmental system, which is a three dimensional system – social, economic and natural – are complex. The most reasonable approach is the use of recycling materials through partial replacement of natural aggregates by recycled concrete aggregate. Unfortunately, direct use of recycled aggregates for production of concrete is accompanied by a number of difficulties. The most important are as follows:

- sharp-edged grains, worsening the workability of concrete mix (however, contrary to that, compressive strength of concrete can be improved);
- increased porosity and water absorption often exceeding 10%, making it impossible to control the behaviour of concrete mix;
- varying strength of parent concrete;
- contamination by soil, clay and heavy metals, which could lead to disorder of hydration processes, especially setting and early stages of hardening (however, at present time this scenario is rather impossible due to application of the pro-environmental procedures in recycling plants).

Despite above mentioned drawbacks, research studies carried out in recent years have shown that recycled concrete aggregate (RCA) successfully can be used for good quality concrete, so long as a number of conditions are met. In practice progress in its application is also observed. For example, in Italy concretes up to C30/37 strength class can contain even 30% of RCA replacing natural aggregate [2].

In the context of the large scale RCA application the main target is to limit water absorption of concrete. It is important for the aggregate not to have high water absorption, although the presence of water in the aggregate before mixing the ingredients could

* Corresponding author.
E-mail address: agra@up.poznan.pl (A.M. Grabiec).

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be beneficial as it leads to self-curing of concrete. For this reason, Tam et al. [3] has recommended a method of double dosing of water (half the volume of water is poured to the aggregate, the rest to the concrete mix), known as two-stage mixing approach (TSMA). The presence of moisture in grains, before adding water, leads to self-curing and might also significantly reduce concrete shrinkage. Studies carried out by Corinaldesi and Moriconi [4] have demonstrated that drying shrinkage of recycled concrete, with water-soaked aggregate as a component, can be about 20–40% less than shrinkage of ordinary concrete.

Nevertheless, lowering water absorption of recycled aggregate is still justified for some reasons. Although the resistance to corrosion process of concrete structures depends mainly on quality of hydrated cement paste, poor quality recycled grains (obtained from inferior quality concrete) to some extent may cause the migration of aggressive media into the microstructure of concrete. Moreover, too porous grains may not retain water before mixing the ingredients, this water is needed for self-curing and is transferred to the cement paste, increasing the value of water to cement ratio in the interfacial transition zone (ITZ) between aggregate and cement paste. If the grains are not moisturised enough, and moisture gradient between recycling grains increases, grains could start absorbing water from the cement paste, causing local drop in the water to cement ratio below value for which complete hydration of cement is possible. Theoretically, w/c = 0.23 is considered as the full hydration limit level, but in practice this value is oscillating around 0.40. Mather and Hime [5] have demonstrated that cement paste cannot contain full hydrated cement when only 0.23 water to cement ratio is provided, because approximately 30% of paste is always occupied by pores created during hydration processes. For complete hydration whole pores volume should be filled by water, despite the fact that substantial part of water does not participate in chemical reactions. However, water consumed by dry recycled concrete grains can considerably weaken ITZ and, as a consequence of it, reduces strength and durability of recycled concrete.

In order to exclude discussed above disadvantages, reduction of water absorption can be achieved by several methods:

– carbonatisation of recycled aggregates – calcite, a product of such carbonatisation, is about 9% bigger by volume than the starting components;
– full or partial water proofing agents – siloxanes, silanes, etc. preparations may be used for this purpose;
– heat treatment, which results in separation of weak mortar in aggregate from the natural grains.

Unfortunately, above mentioned methods are burdened with a number of disadvantages. Carbonatisation is a slow process, and since in practice recycling grains are usually quickly put into new applications, therefore, they do not have time to undergo carbonatisation. Chemical substances are used for water proofing but this is not compatible with sustainable development principle. Heat treatment comes with additional energy input, which is also not beneficial from the environmental perspective.

The method of biodeposition of calcium carbonate conducted through the participation of *Sporosarcina pasteurii* bacteria should constitute an alternative method. Biodeposition, as opposed to other concepts, is a natural method and, in principle, makes less severe with the environment, because all the components used for cultivating the substrates as well as and the strain itself, naturally occur in the environment [6]. The biodeposition concept is based on the ability of bacteria to precipitate calcium carbonate on the outer surface of the cell wall, due to occurrence of negative zeta potential of adequate strength. Biodeposition process has been described as follows [7]:

$$Sp.cell + Ca^{2+} \rightarrow Sp.cell - Ca^{2+} \quad (1)$$

$$CO(NH_2)_2 \xrightarrow{urea} 2NH_4^+ + CO_3^{2-} \quad (2)$$

$$Sp.cell - Ca^{2+} + CO_3^{2-} \rightarrow Sp.cell - CaCO_3 \downarrow \quad (3)$$

S. pasteurii cell (*Sp. cell*) can attract Ca ions (Ca^{2+}), which react with carbonate ions (CO_3^{2-}) originating from urea ($CO(NH_2)_2$) hydrolysis. Simultaneously, ammonia ions (NH_4^+) increase pH value in surrounding medium which improves calcite precipitation efficiency [8].

Biodeposition method has already found practical application in civil engineering, as reported by, among others, Czarnecki and Lukowski [9], Wiktor et al. [10], De Muynck et al. [11,12], Van Tittelboom et al. [13]. The concept of microbiological method of precipitation of calcium carbonate has also been experimentally verified in other areas (e.g. carbon capture and storage technologies) [14].

2. Materials and methods

2.1. Recycled concrete aggregate

Recycled aggregate was obtained from the concrete, made from Portland cement CEM I 42.5 R and natural aggregate, produced in commercial concrete plant. Concrete mix compositions are presented in Table 1. The average values of a 28-day compressive strength of concrete, for the water to cement ratio w/c = 0.45, was −42.2 MPa, and for w/c = 0.70 −17.4 MPa. After 56 days, concrete was crushed in a commercial crushing plant (EKO-ZEC Poznan), transported to the laboratory and subjected to separation into fractions by sieving through a set of sieves with meshes measuring: 1, 2, 4, 6, 8, 12, 16 and 19 mm. Simultaneously the average density of RCA was determined on the base of parent concrete density. It was equal to 2333 kg/m^3 for both values of w/c.

After 2 years of storage in an internal environment, temperature: 20 ± 2 °C and relative humidity RH about 60%, some of the aggregate grains were re-crushed in a laboratory jaw crusher, in order to compensate the shortage of quantity of some fractions. Aggregate with 6/8 mm and 12/16 mm fractions were used for subsequent tests. The remaining fractions were used for other tests.

2.2. Bacterial strain and culture media

Bacterial strain called *S. pasteurii* (previously known as *Bacillus pasteurii*), No DSM 33, bought from the German Collection of Microorganisms and Cell Cultures (DSMZ) in Braunschweig was used for the tests. This strain was chosen because of its greatest potential for precipitation of calcium carbonate, even under extreme conditions, as well as its lack of pathogenicity.

The strain was revived on agar slants with the following composition: meat extract (3 $g\,l^{-1}$), peptone (5 $g\,l^{-1}$), urea (20 $g\,l^{-1}$) and agar (15 $g\,l^{-1}$), prepared on deionised water to avoid the influence of contaminating ions [6]. After activation of bacteria, they were inoculated with liquid culture medium deprived of urea, inoculum resulting from the agar slants wash. The final density was 10^{13}–10^{14} cells ml^{-1}. The liquid culture, in polypropylene containers, sealed using cellulose stoppers that allow for gas exchange, were kept for 24 h in an incubator, with shaking (170 rpm) at temperature of 30 °C. The shaking was applied to improve aeration and guarantee uniform development of the culture medium.

Concentration of the culture marked in Thoma counting chamber was 10^7–10^8 cells ml^{-1} of culture medium. Spectrophotometric analysis of the culture liquid showed that absorbance rate was 0.562, at a wavelength of 430 nm and 0.362 at a wavelength of 600 nm.

S. pasteurii culture kept under these conditions for 24 h was next moved to the concrete laboratory and poured into glass containers with recycled aggregate samples.

2.3. Whey as alternative culture medium

Strain of *S. pasteurii* was also used in the additional test. Cultivation of studied microorganisms was carried out on the substrate similar to the one suggested by Achal et al. [6], containing: whey (10% w/v, of composition given in Table 2), NaCl (5 $g\,l^{-1}$), urea (20 $g\,l^{-1}$) and agar (15 $g\,l^{-1}$). Control culture medium was substrate containing meat extract and peptone with the following constituents: meat extract (3 $g\,l^{-1}$), peptone (5 $g\,l^{-1}$), urea (20 $g\,l^{-1}$) and agar (15 $g\,l^{-1}$).

The cultivation was carried out under stationary conditions at 37 °C for 48 h. Microorganisms count was determined using a plate method.

Table 1
Recipes of parent concretes used for production of recycled aggregate.

w/c (–)	Cement (kg/m^3)	Water (kg/m^3)	Sand (kg/m^3)	Gravel	
				2/8 mm (kg/m^3)	8/16 mm (kg/m^3)
0.45	405	183	615	601	601
0.70	290	146	706	689	689

Table 2
Composition of whey.

Component	Unit	Content
Solid residue	% (weight)	90.20 ± 0.28
Ash	% (w)	8.80 ± 0.13
Proteins	% (w)	10.810 ± 0.137
Fats	% (w)	0.260 ± 0.014
Carbohydrates		
DP3	% (w)	0.065 ± 0.011
Lactose	% (w)	81.82 ± 3.80
Galactose	% (w)	17.13 ± 0.034
Calcium (EDTA)	mg/g	17.9 ± 1.4
Calcium (ASA)	mg/g	16.80 ± 0.57

2.4. Water absorption test

A generally recognised method, according to EN 1097–6 standard (*Tests for mechanical and physical properties of aggregates. Determination of particle density and water absorption*), applied for ordinary aggregates, modified in terms of drying temperature, was used to determine the water absorption of recycled aggregates. Instead of the temperature being kept at 105 ± 5 °C, it was maintained at 78 °C in order to avoid removal of interpacket water from hydrated calcium silicates (CSH), which occurs at 78–90 °C [15] and which could have skewed the results.

The dried aggregate samples were placed in picnometers and closed tightly. Next, distilled water was poured, and by slowly turning and shaking air was removed from the pore space. Filled picnometers were left for 24 h. After this time, the process of turning and shaking was repeated. Water was poured out, the aggregate was predried and subsequently water drained from grains of aggregate on a sieve of sieve mesh size 0.4 mm. Then, the aggregate was transferred to dry cotton cloths, arranging the samples of aggregates in such a way that the grains did not cling to each other. Transfer to subsequent dry cloths was continued until the aggregates lost visible surface water film. The samples of aggregates were placed in glass containers and weighed. From the value of weight of dry aggregate and weight of moistened aggregate, the water absorption was determined. The number of samples needed to determine the water absorption of aggregates in the test was five.

2.5. Biodeposition treatment procedure

Liquid culture of *S. pasteurii* was poured to each of the aggregate samples dried at 78 °C, and left for 24 h. Aggregate samples on which water absorption tests were carried out before biodeposition were used.

Calcium carbonate was precipitated as a result of hydrolysis of urea and adsorption of calcium ions on the bacteria cell surface. The calcium salt solution, which was poured after the above-specified period was prepared using deionised water, urea and calcium chloride to yield 2% (by mass) urea and calcium ion concentration equal 0.3 mol l^{-1} [7] in the entire volume of solution in the third stage (stage 3), which has been shown in Fig. 1. Calcium chloride was used for its better solubility and better accessibility of calcium ions in a biodeposition process. Undoubtedly, some other calcium salts (e.g. acetate and nitrate) could be possible to apply in practice and be safer for reinforced concrete structures [13].

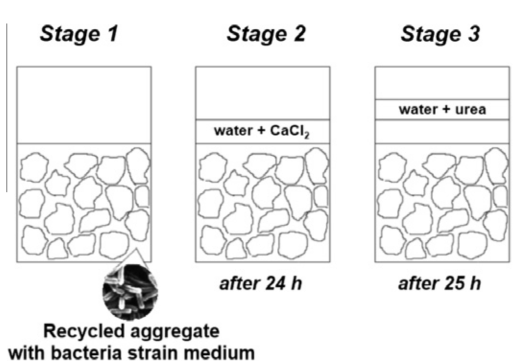

Fig. 1. Procedure of biodeposition experiment.

The volume of solution in the second phase (stage 2) was 15% higher than in the first phase (stage 1). The samples were kept under a constant temperature of 26 °C. After 5 days, the reaction solution was removed, and samples were once more dried at 78 °C in order to prepare them for re-test for water absorption.

2.6. Statistical analysis

An analysis of statistical results of water absorption of aggregates was carried out. A hypothesis on the difference of mean values of two normal distributions, under the assumption of independence of variables and the adoption of Student's t-distribution was examined. Statistical calculations were performed using *Statistica* (Software program, licence no: JGNP 105B037825 AR-A). Statistical null hypothesis was formulated, that assumed that there is no mean difference between the absorption values before and after application of bacteria. The validity of the hypothesis at two *p*-levels (error significance levels): 0.05 and 0.10 was checked.

2.7. SEM analysis

Samples of 12/16 mm aggregate fractions obtained from crushed concrete, with water to cement ratio w/c = 0.45, were analysed under ZEISS 435 VP scanning electron microscope, before and after biodeposition. Preparations from dried aggregate grains covered with a thin layer of gold were used. Observations were made at accelerating voltage 15 kV and working distance (WD) 6 mm. The study was qualitative in nature, its objective was to confirm the phenomenon of calcium carbonate precipitation.

3. Results and discussion

The results of research on water absorption of recycled aggregates before and after modification with the use of *S. pasteurii* have been shown in Fig. 2.

Biodeposition of aggregate resulted in a slight decrease in its water absorption, greater in the case of aggregate coming from concrete with the w/c = 0.70 and 28-day compressive strength equal 17.4 MPa compared with aggregate from concrete with w/c = 0.45 and 28-day compressive strength of 42.2 MPa (Table 1). This leads to conclusion that better results in terms of biodeposition efficiency are obtained for aggregates from concrete of lower compressive strength. This offers the opportunity to improve the

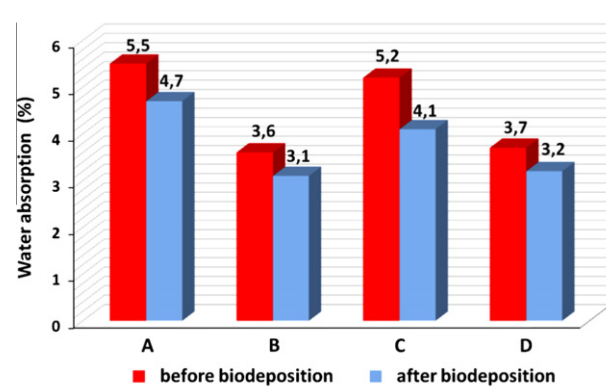

Fig. 2. Water absorption of recycled concrete aggregate (average values) before and after biodeposition (A – w/c = 0.45, fraction 6/8 mm; B – 0.45, 12/16; C – 0.70, 6/8; D – 0.70, 12/16).

Table 3
Efficiency of whey as an alternative culture medium.

Designation of culture	Bacteria quantity (cfu ml^{-1})
1	$3,7 \times 10^5$
2	$1,2 \times 10^5$
3	$3,2 \times 10^5$
4	$9,0 \times 10^6$

Culture 1–3: whey as a medium; culture 4 (control): standard medium.

quality and increase the use of lower quality recycling aggregates. Better water absorption was observed in the case of aggregates of finer grain size, confirming an average of 30–35% better water absorption than before biomodification and 50% greater drop in water absorption for aggregates of particle size 12/16 mm, after biodeposition. The general trend to lower water absorption of aggregates through the use of *S. pasteurii* shows that bacteria have a positive impact on this property of aggregates. In the research studies conducted, drop in water absorption rate was not particularly spectacular, however, this in the least does not diminish their implication. Nevertheless, it has to be stressed that the time between the moment of crushing of concrete and of testing the aggregate from this concrete is factor that could have contributed to attaining such results. This time was 2 years. Due to high surface area of the resultant crushed particles, crushing of concrete accelerates carbonatisation process, which was confirmed by Ajdukiewicz and Kliszczewicz [16]. Earlier unpublished studies conducted by the authors of this paper, have also shown that carbonatisation implemented using accelerated method decreases the water absorption of aggregates by between 10% and 20%. It is believed that only slight decrease in water absorption by precipitation of calcium carbonate in a microbiological way was due to carbonatisation that took place before the start of water absorption experimental measurement. Calcium carbonate in form of solid precipitate weakened the migration of water deep into the aggregate, hence reducing the effectiveness of process. It could, therefore, be expected that biodeposition process carried out on the aggregate immediately after crushing would lead to better efficiency. In order to test this hypothesis, through determination of the true biodeposition potential, unaffected by carbonatisation, it is advisable to carry out further research studies, focusing on analysis of aggregate derived directly from crushed concrete under fully controlled conditions. At the same time it is worth emphasising that this study is only a contribution to describe how biodeposition improves the quality of concrete made of RCA. It is necessary to do experimental work on compressive strength and durability of recycled aggregate concrete. In this context worth following seem to be studies of Tam et al. [17], Tam and Tam [18], Tam et al. [19] on both mentioned above properties of recycled aggregate concrete. Two-stage mixing approach has been proposed to improve strength, water, air and chloride permeability of RA concrete [17,19]. Pre-soaking the RCA has been also concluded to be an effective method to improve mechanical properties of the concrete made of recycled concrete aggregate [19]. Undoubtedly, strength and durability characteristics of RA concrete can be also remarkably improved by adding superplasticisers to reduce water to cement ratio and as a result to obtain a denser concrete [20,21].

The highest concentration of microorganisms tested (approximately 10^6 cfu ml^{-1}) was recorded after use of culture medium containing meat extract and peptone (Table 3). On a substrate with whey, the number of cells after 48 h of incubation, on average, was 10^5 cfu ml^{-1}, which confirms the usefulness of whey as an alternative culture media for *S. pasteurii*. According to Whiffin [22] brewery waste yeast and torula yeast, being the wastes from agriculture and food industry, can be also used as an alternative to expensive protein sources (yeast extract). It has been demonstrated that the brewery waste yeast and torula yeast (minimum 54% of proteins) application cost is 20-fold lower (9$ Australian dollars per 1 kg) and 100-fold lower (2$ per 1 kg), respectively, in comparison to the cost of yeast extract. Although two mentioned sources contain less proteins than the latter (66% of proteins), this replacement can be promising alternative prospect of biodeposition technologies.

The biodeposition phenomenon on the surface of aggregate grains has been confirmed by observations under scanning electron microscope (Figs. 3 and 4). Qualitative observations under SEM showed clear presence of crystals of calcium carbonate, on the amorphous microstructure of the material, in the aggregate samples subjected to biodeposition – it was not present in samples not exposed to bacteria action. In the present study, calcium chloride of one concentration was used as a salt, whose presence causes precipitation of calcium carbonate. Hence, the effect of the type of anions, concentration of calcium salt as well as other factors such as temperature and relative humidity RH at the biodeposition process were not analysed. Research studies aimed at determining the effect of these factors on polymorphic modifications of resultant calcium carbonate would certainly be worth expanding, especially employing other instrumental methods, for example, X-ray analysis. According to literature reports, change of concentration of calcium salt used in biodeposition results in modification of the morphology of precipitated calcium carbonate [10].

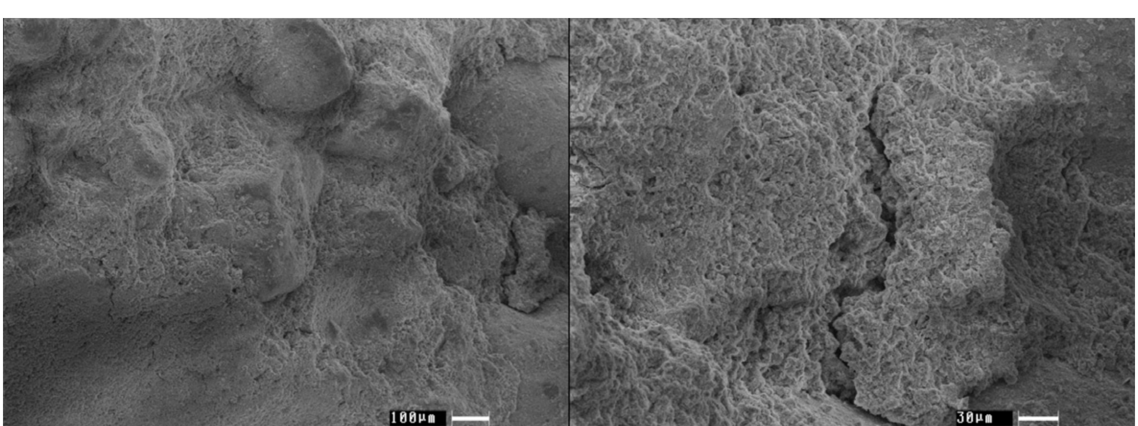

Fig. 3. Scanning electron micrographs of recycled aggregate grain (w/c = 0.45, fraction 12/16 mm) without biodeposition treatment (magnification: left – 150×, right – 536×).

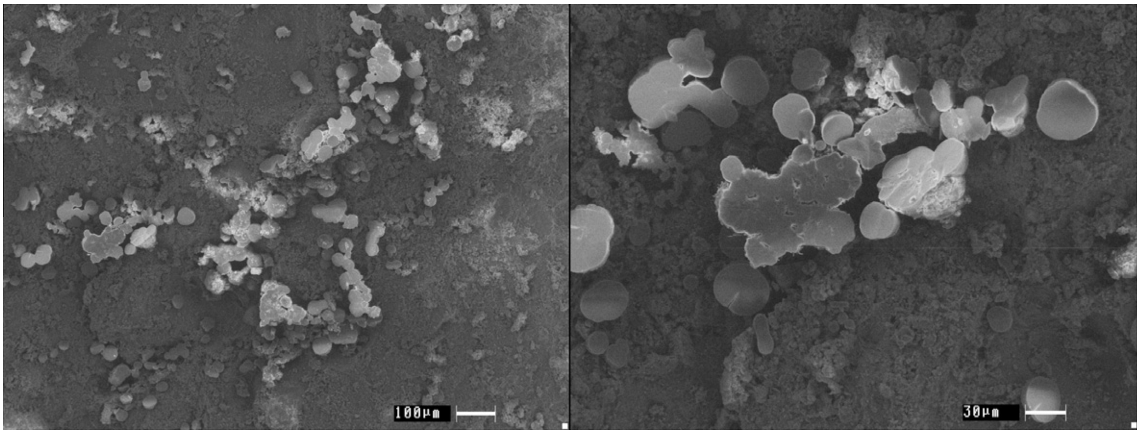

Fig. 4. Scanning electron micrograph of recycled aggregate grain (w/c = 0.45, fraction 12/16 mm) after biodeposition treatment (magnification: left – 158×, right – 545×).

Table 4
Statistical test results of recycled aggregate water absorption before and after biodeposition for error significance level of 0.05 and 0.10.

Error significance level	0.05	0.10
Confidence level	0.95	0.90
Series	H_0 hypothesis test	
A	Rejected	Rejected
B	Rejected	Rejected
C	Rejected	Rejected
D	Accepted	Rejected

H_0 – lack of statistically significant difference between each series before and after biodeposition.

Based on statistical analysis, consisting in test for significant differences between the means of two series (before and after biodeposition), assuming an hypothesis that there is no statistical significance, it was found out that lowering of water absorption rate was statistically significant at p-level of 0.10 for all test series (different values of w/c and aggregate fractions) and at p-level of 0.05, three cases out of four. The results are presented in Table 4.

4. Conclusions and perspectives

Used in the research studies, the method of biodeposition of calcium carbonate seems to be a promising technique in the improvement of the quality of recycled aggregate. It led to reduction in the water absorption of aggregate and this was even more effective when finer fractions derived from inferior quality concrete were used. The biodeposition process unambiguously was confirmed by the observations in SEM. The method, as it is still little known and recognised, should be scientifically as well as practically developed. This requires interdisciplinary approach of two scientific branches that are a far apart: concrete technology and microbiology, which results in need for closer cooperation between the two fields in order to overcome existing scepticism. There are many research aspects worth deeper research, such as type and concentration of calcium salt, whose presence leads to precipitation of calcium carbonate, the temperature and relative humidity, type of culture media, including the possible use of recycled materials, seeking extremophile types of bacteria or creating them due to genetic modifications. The work is a contribution to subsequent research studies on modification of properties of recycled materials in compliance with environmentally sustainable development. After refining the methods and possible subsequent implementation, there is a chance for greater use of recycled concrete aggregates, at least in low and medium strength concrete.

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