

Selected properties of concrete with recycled aggregate subjected to biodeposition

A.M. GRABIEC^{1*}, D. ZAWAL¹, J. STARZYK², and D. KRUPA-PALACZ¹

¹ Poznań University of Life Sciences, Institute of Construction and Geoengineering, 94E Piątkowska St., 60-649 Poznań, Poland

² Poznań University of Life Sciences, Department of Agricultural Microbiology, 50 Szydlowska St., 60-656 Poznań, Poland

Abstract. In this paper, the influence of biomodification of recycled concrete aggregate (RCA) on some properties of concrete was studied. *Sporosarcina pasteurii* strain was chosen for biodeposition process. The RCA came from parent concretes with varying w/c ratio. Recycled aggregate concrete (RAC) with two levels of w/c ratio, made from RCA not subjected to biomodification, was treated as reference. Compressive strength, water absorption and sorption of concretes were tested. The most significant influence of the aggregate biomodification was found in the case of sorption and this effect was highest for RAC made from the aggregate yielding from better quality parent concrete.

Key words: recycled concrete aggregate, concrete properties, biodeposition.

1. Introduction

Sustainable development in civil engineering implies the requirement of searching for environment-friendly technological and engineering solutions. An interdisciplinary approach to scientific research is very helpful in this context. The scope of analysis of biomodification of cement matrix composites involves both the fields of concrete technology and microbiology.

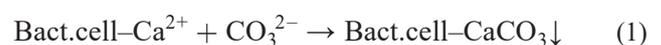
Recycled concrete aggregate (RCA) obtained from dismantling and reconstruction works on concrete and reinforced concrete structures is a serious burden to the environment. Assuming, as in [1], that concrete constitutes 40% of civil engineering waste and that this concrete can be crushed in total, then globally in the near future we will be facing the task of recycling about 1 Gt waste in the form of RCA. Therefore, its rational recycling in civil engineering forms a problem of sustainable development. Although according to some opinions “waste must be prevented rather than limited and or even recycled, in order for construction to produce positive inputs (...)” [2], by-products are still used for limiting their amount and much time is needed to obtain “the goal being a zero waste system”.

Many studies have been devoted to RCA. One of the most recent and comprehensive sources is [3]. The authors have presented a review of 2000–2017 literature on the production and utilisation of recycled aggregate in civil engineering. Such a review may undoubtedly promote the use of recycled concrete aggregate; in fact, such applications are increasingly popular as a part of sustainability. However, RCA’s impact on civil engineering understood as obvious replacement of some materials

by recycled aggregate is impossible to generalise. It depends on the economy of each country, sufficient knowledge and regulatory frameworks. Hence differences arise between developed and developing countries.

One of possible uses for RCA is to replace with it some amount of natural aggregates used in concrete production. This has to be urgently undertaken, because we are running out of deposits of natural aggregate, especially gravel. Its excavation leads to severe degradation of landscape and even to lowering of ground water level. However, the use of RCA is not rational without improving some of its properties, since it is usually of poor quality (large porosity, water absorption, water demand and content of irregularly shaped grains) and lack of uniformness [4–8]. RCA is to be a constituent of concrete in service in various environmental conditions. According to sustainable development in civil engineering, it has to fulfil special requirements related to strength and durability. That is why the properties of RCA have to be modified. It is done by removing old cement mortar or cement paste by: heating and rubbing [9, 10], ultrasonic cleaning [11], mechanical grinding, gravity classification [5, 7] and acid cleaning [12, 13]. Alternatively, old cement mortar may remain in RCA but it has to be strengthened using polyvinyl alcohol, nano-materials [14, 15] or by carbonation [6, 8, 16–18].

A relatively new method proposed for recycled concrete aggregate modification is biomineralisation. This approach was motivated by a significant improvement observed in stone materials, natural and artificial – mortars and concretes [19–25], as well as some soils [26–33]. The methods use the process of microbiologically driven production of calcium carbonate, based on the ability of bacteria to precipitate calcium carbonate on the outer surface of the cell wall (1), due to negative zeta potential of adequate strength [34]:



*e-mail: anna.grabiec@up.poznan.pl

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Bacteria cells attract Ca^{2+} ions, which react with carbonate ions CO_3^{2-} originating from urea hydrolysis $\text{CO}(\text{NH}_2)_2$. Ammonia ions NH_4^+ increase pH value in surrounding medium which improves calcite precipitation efficiency [35].

The wider and wider interest in the biomineralisation method as an ecological means of improving some mineral materials properties is reflected in overview papers summarizing the current state of art on the international level [8, 22, 36–41].

A comprehensive approach to this problem, taking into account biochemical and biotechnological aspects, is presented in monograph [42]. Clearly, the problem is of great importance.

First papers devoted to the use of biodeposition in order to improve properties of recycled concrete aggregate date back to 2012 [43] and 2014 [44]. In both papers the authors focused on an especially important property of RCA, i.e. water absorption. It was found that biodeposition of RCA resulted in a greater decrease of its water absorption in the case of aggregate coming from parent concrete of lower compressive strength and in the case of finer grain size aggregate. Observations under scanning microscope confirmed the presence of crystals of precipitated calcium carbonate in aggregate samples subjected to *Sporosarcina pasteurii* action [43] which, according to [44], are similar to vaterite.

Newer research [45, 46] concerns the biomodification of RCA by *Bacillus sphaericus*. It was concluded that properties of resulting concrete, like water absorption, modulus of elasticity and compressive strength, were slightly better, and in the least satisfactory cases they were comparable to the properties of concrete made with aggregate not subjected to biomineralization. Potential of *Bacillus sphaericus* was also emphasized by [47] in the biomodification of ceramic recycled aggregate.

With regard to economic aspects it is too early to estimate or even predict financial benefits coming from biodeposition of RCA. Studies presented in literature are mainly focused on scientific aspects. Too few experiments have been carried out and too many questions are still open. Moreover, as mentioned in relation to the overall impact of recycled aggregate on civil engineering, quite a lot depends on the economic growth of each country. In addition, there is another barrier that limits the practical implementation of recycled concrete technology in general, which is correlated with the transfer of scientific achievements to industrial practice. In this paper the authors combine the technological and microbiological approach.

The aim was to find out whether biomineralisation of recycled concrete aggregate influences some properties of concrete. Properties of concrete mixes and hardened concretes, in particular compressive strength, water absorption, and sorption were described. The two latter properties were considered as indicators of concrete durability, which is one of the main scientific aspects of concrete technology and has started to develop particularly intensively over the last two decades [48].

2. Materials and methods

2.1. Aggregate. Recycled concrete aggregate was prepared in laboratory crusher of two parent concrete series with dif-

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

Concrete w/c (-)	Cement (kg/m ³)	Water (kg/m ³)	Sand (kg/m ³)	Gravel (kg/m ³)	
				2/8 mm	8/16 mm
0.45	400	180	596	741	494
0.65	250	163	653	812	541

ferent w/c ratio (0.45 and 0.65). The composition of parent concretes is presented in Table 1. The sand and gravel used were obtained from local deposits. The 28-day compressive strength was 50.6 MPa and 26.9 MPa for w/c ratio 0.45 and 0.65, respectively. The same sand as for parent concretes was used as fine aggregate to prepare recycled aggregate concrete (RAC). Coarse aggregate was adopted in the form of RCA divided into fractions: 2/4, 4/6, 6/8, 8/12.3 and 12.3/16 mm fulfilling the concept of Fuller ideal grain composition. The equation used is shown as follows:

$$P = (d/D_{max})^n \tag{2}$$

where P is content of aggregate grains with dimension smaller or equal to d; d is the grain dimension smaller or equal to d value (mm) resulting from the Fuller's curve; D_{max} is the maximum aggregate dimension (mm) in the entire aggregate volume composition (in our case 16 mm); n is the specific coefficient of gradation, usually assumed as equal to 0.5.

2.2. Cement. In both cases, for parent concrete and recycled aggregate concrete (RAC), Portland cement CEM I 42.5 R, manufactured by LafargeHolcim in the cement plant Kujawy in Bielawy, Poland, was used. Properties of the binder are shown in Table 2. The pure-clinker neutral type of binder was chosen due to the character of experiment, in order to eliminate any possible influence of mineral additives present in the cement paste on the hydration process.

Table 2
Physical, mechanical and chemical properties of cement

Characteristic	Result	
Blaine's specific surface (m ² /kg)	343	
Initial setting time (min)	146	
Final setting time (min)	190	
Density (kg/m ³)	3.09	
Compressive strength (MPa)	2 days	28.8
	28 days	54.1
SO ₃ (%)	3.22	
Cl ⁻ (%)	0.069	
Na ₂ O _{eq} (%)	0.61	

2.3. Superplasticiser. The highly effective fluidifying admixture of a new generation – polycarboxylate ether superplasticiser for RAC mixes – was used in order to improve workability. The properties of the admixture are: pH – 6, specific density – 1.07 kg/dm³, solid content – 30%, Na₂O_{eq} ≤ 0.8%.

2.4. Bacterial strain and culture media. *Sporosarcina pasteurii* strain (previously known as *Bacillus pasteurii*) was chosen for biomodification because of its great potential for precipitation of calcium carbonate, even under extreme conditions, as well as the lack of pathogenicity. This strain is frequently used to improve quality of mineral materials. The strain proved to be effective in experiments [33, 43]. The strain was supplied by the German Collection of Microorganisms and Cell Culture in Braunschweig (designated as DSM 33) and was originally isolated from the soil.

Sporosarcina pasteurii was revised on agar slants with the following composition: meat extract (3 g/dm³), peptone (5 g/dm³), urea (20 g/dm³) and agar (15 g/dm³) prepared on deionised water to avoid the influence of contamination ions [49]. After activation of bacteria, they were inoculated with liquid culture medium deprived of urea, inoculum resulted from the agar slants wash. The liquid culture in polypropylene containers, sealed using cellulose stoppers that allow for gas exchange, was kept for 24 hours in an incubator, subjected to shaking (170 rpm) at the temperature of 30°C. The shaking was applied to improve aeration and to guarantee a uniform development of the culture medium. *S. pasteurii* culture kept under such conditions for 24 hours was then moved to concrete laboratory to be used for biodeposition process.

2.5. Water absorption test. A generally known method, according to EN 1097–6:2013 standard. *Test 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 water absorption of each recycled aggregate fraction. The number of replicates for each fraction was three. The measurements were made for cognitive purpose and to verify the final recipes for RAC in the context of excess water (resulting from water absorption of RCA), necessary to counteract the effect of sorption of effective water from paste by recycled concrete aggregate (which could lead to a more dense consistency of a concrete mix). Instead of the temperature being kept at 105±5°C, it was maintained at 77°C in order to avoid removal of interpacked water from hydrated calcium silicates (C-S-H), which occurs at 78–90°C [50] and could influence the results. The dried aggregate samples were placed in pycnometers and closed tightly. Next, tap water was poured, and slow turning and shaking was repeated. Submersion of RCA in water lasted for 30 minutes. Water was poured out, the aggregate was pre-dried and, subsequently, water was drained from grains of aggregate on a sieve of the mesh size 1.0 mm. Then, the aggregate was transferred to dry cotton cloths, the samples of aggregate were arranged in such a way that grains did not cling to each other. Subsequent transfer to dry cloths was continued until the aggregate lost a visible surface water film.

The number of samples used to determine the water absorption of aggregate in the test was three.

2.6. Biodeposition treatment procedure. In the case of RCA biomodification, a liquid culture of *Sp. pasteurii* was poured to each of aggregate fraction samples dried at 77°C (the value equivalent to the temperature in the water absorption test) and left for 24 hours. Aggregate samples on which the 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 bacteria cell surface. The calcium salt solution, which was poured after the above mentioned period, was prepared using deionised water, urea and calcium chloride to yield 2% (by mass) urea and calcium ion concentration equal 0.36 mol/dm³ in the entire volume of solution. Calcium chloride was used for its better solubility and better accessibility of calcium ions in the biodeposition process. The samples were kept under a constant temperature of 23°C for 7 days. In order to allow for a better oxygen access and bacteria activation, containers with biomineralized aggregate were shaken on a daily basis.

2.7. Recipes and preparation of basic concrete mixes. Recipes for RAC mixes (Table 3) were established basing on recipes of parent concretes. Amounts of particular fractions of RCA were adopted to replace gravel fractions, water amount was modified taking into account the water absorption of RCA equal to 3.8% as the weighted mean value for RCA from the parent concrete with w/c ratio 0.45 and 4.4% for RCA from the parent concrete with w/c ratio 0.65. Superplasticiser was used to achieve required fluidifying effect of the concrete mixes.

Table 3
Recipes of recycled aggregate concretes

Constituent	Recipe designation (and w/c ratio of parent concrete of RCA)			
	R-1 (0.45) R-2 (0.65)	R-3 (0.45) R-4 (0.65)	R-5 (0.45) R-6 (0.65)	R-7 (0.45) R-8 (0.65)
	Unmodified		Biomodified	
	RAC_0.45	RAC_0.65	RAC_0.45	RAC_0.65
Effective w/c	0.45	0.65	0.45	0.65
Cement	370	250	370	250
Superplasticiser	3.7	2.5	3.7	2.5
Sand	686	724	686	724
RCA*				
2/4 mm	246	260	246	260
4/6 mm	189	199	189	199
6/8 mm	159	168	159	168
8/12.3 mm	284	299	284	299
12.3/16 mm	209	220	209	220
Effective water	166.5	162.5	166.5	162.5
Additional water**	41.2	51.0	41.2	51.0
Total amount of water	207.7	213.5	207.7	213.5

* Recycled concrete aggregate

** Calculated taking into account RCA water absorption.

The constituents of concretes were mixed using a paddle-type 0.05 m³ mixer. Mixes were prepared using the two stage mixing approach (TSMA) proposed by [51]. The method involves adding 50% of water only with the aggregate leaving it for 30 minutes to saturation, while the remaining water is added in a traditional manner. According to those authors such an approach improves significantly the concrete quality. After a 30-minute break cement was added and the composition was mixed for a 45-second period. Then the remainder of water with superplasticiser was added and mixed for a 3-minute period.

The compressive strength tests were conducted on 100 mm cubic samples. The results were recalculated to the case of 150 mm cubic samples and these ones were analysed further. Testing of compressive strength was carried out according to the European standard EN 12350-3:2009. *Testing hardened concrete – Part 3. Compressive strength of test specimens*. After demoulding the samples were cured in water at 20°C until the time of testing – 28 days. The number of samples for each examined series of concrete was five.

The water absorption was evaluated using 100 mm concrete cubes (three for each examined series) at the age of 28 days as the ratio between the maximum water mass absorbed by concrete samples and the mass of samples dried at the temperature of 105°C. The procedure conformed to the Polish standard PN-88/B-06250. *Ordinary concrete*.

Testing of sorption as a useful parameter for assessment of concrete durability is proposed, among others, by [52] and alternatively by the European standard EN 13057:2002. *Products and systems for the protection and repair of concrete structures. Test methods. Determination of resistance of capillary absorption*. In this research a combined method of sorption testing was used – thickness of samples and the temperature followed [52] but diameter of samples and procedure conformed to standard EN 13057:2002. Moreover, time of measurement was prolonged to 72 hours and the measurement after 72 hours provided additional result. Both methods are based on an assessment of a mass change for water saturated samples after capillary absorption for up to 72 hours. 28-day-old cylindrical samples with 100 mm diameter and 25 mm height (nine per series) with side walls protected by a silicon mass were dried at 50°C for 5 days. In the subsequent stage they were put onto a plastic mesh to allow for a free water absorption on the entire base surface without a direct contact with the container bottom. Water level was equal to 1/5 of the sample height. Measurements were performed after: 15, 30, 45, 60 minutes and then after: 2, 3, 4, 6, 9, 24, 48 and 72 hours in order to evaluate the rate of sorption.

Statistical analysis of the results was done using the measurements after 72 hours in relation to the initial mass. *Statistica* software, license No JGNP 105B037825 AR-A from Poznań University of Life Sciences was used. The following analyses were carried out:

- variance for factor sets assuming three input variables, each on two levels:
 - w/c ratio of parent concrete samples (from which RCA was obtained) – $RCA_{w/c}$ (0.45 and 0.65 – RCA_{45} and RCA_{65}),

- water to cement ratio of RAC – $RAC_{w/c}$ (0.45 and 0.65 – RAC_{45} and RAC_{65}),
- aggregate type (unmodified and modified by biodeposition – *unmodified* and *biomodified*, respectively),
- post-hoc test (Scheffe test).

Statistical analysis concerned compressive strength and simplified sorption test results. It was possible because of the above mentioned number of replications (five and nine, respectively). One has to note, that the use of the variance analysis is possible under the condition of normal distribution. In the case of compressive strength this property is commonly known. In the case of the simplified sorption test, a verification of the hypothesis of the normal distribution was carried out first. Shapiro-Wilk test was applied to this end. Water absorption testing was carried out with three replications, which did not allow for the variance analysis.

Scheffe test was chosen due to its conservative character, what means that the differences between series are more difficult to be assumed as significant when compared to other tests (e.g. Tuckey test). The use of that test is justified due to the specific nature of recycled concrete aggregate, featuring nonuniformity of the composition with a varying proportion between natural and mortar-covered grains.

3. Results

Results of consistency testing of concrete mixes with air content not exceeding 3.6%, both biomodified and prepared with aggregate not subjected to biodeposition, are presented in Table 4. Concrete mixes on biomineralised aggregate featured a more dense consistency than those on aggregate not subjected to biodeposition. Similar results were reported by [45], who used *Bacillus sphaericus* bacteria to modify RCA and calcium nitrate as a source of calcium ions.

Table 4
Consistency of concrete mixes from recycled concrete aggregate

water/cement ratio		Slump (mm)			
RAC (concrete)	RCA (aggregate)	Unmodified aggregate		Biomodified aggregate	
0.45	0.45	R-1	45	R-5	35
0.45	0.65	R-2	25	R-6	10
0.65	0.45	R-3	13	R-7	0
0.65	0.65	R-4	9	R-8	1

Results of compressive strength clearly confirmed the obvious fact that RAC with lower w/c ratio features higher values of compressive strength. Analysis of Fig. 1 indicates a decrease of strength in the series of concretes with RCA subjected to biomodification, in which better quality aggregate was used with w/c equal to 0.45 as compared to the reference concrete – decrease of 3.1 MPa and 3.4 MPa for concrete with w/c ratio 0.45 and 0.65, respectively. However, in the statistical analysis

Table 5
Compressive strength of concretes made from RCA, unmodified and subjected to biodeposition in Sheffe test

recipe no. Avg c.s.	Designation of series (recipe no.) Average compressive strength (MPa)						
	R-1 53.36	R-2 33.44	R-3 48.13	R-4 27.94	R-5 50.30	R-6 30.00	R-7 47.88
Unmodified							
R-1 53.36							
R-2 33.44	0.00000						
R-3 48.13	0.00061	0.00000					
R-4 27.94	0.00000	0.00028	0.00000				
Biomodified							
R-5 50.30	0.13785	0.00000	0.53758	0.00000			
R-6 30.00	0.00000	0.06223	0.00000	0.60279	0.00000		
R-7 47.88	0.00031	0.00000	0.99999	0.00000	0.39809	0.00000	
R-8 28.78	0.00000	0.00292	0.00000	0.99529	0.00000	0.95851	0.00000

(Table 5) it was proven that these differences are not statistically significant. A similar lack of statistical dispersion was found for the series with RCA of a worse quality (from the parent concrete with $w/c = 0.65$). It is evident in Fig. 1 (differences in mean values amount to 0.2 MPa and 1.1 MPa for RAC with w/c ratio of 0.45 and 0.65, respectively). Comparison between series of concretes including the aggregate subjected to biodeposition did not reveal any statistical dispersion between series with different RCA but resulting from the same recipes. A similar comparison of control series (not subjected to biomodification)

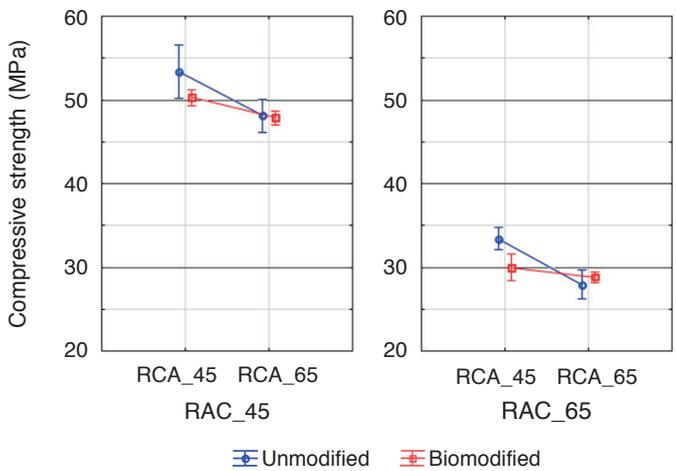


Fig. 1. Compressive strength of concrete made from RCA

Table 6

Water absorption of concretes from recycled concrete aggregate

water/cement ratio		Water absorption (%)			
RAC (concrete)	RCA (aggregate)	Unmodified aggregate		Biomodified aggregate	
0.45	0.45	R-1	7.4	R-5	7.3
0.45	0.65	R-2	7.6	R-6	7.4
0.65	0.45	R-3	7.7	R-7	7.6
0.65	0.65	R-4	8.9	R-8	8.8

did show a statistical dispersion. Thus, it can be concluded that biomodification equilibrates concrete strength independently of the RCA quality.

Water absorption of concrete (Table 6) was high (between 7.3% and 8.9%), and it concerns both concrete types – unmodified and subjected to biodeposition. It is due to the high content of RCA in concrete (about 65% of aggregate volume – all fractions over 2 mm were exclusively RCA). The influence of biomodification led to a minimal decrease of water absorption (the largest change of 2% was noted for the case with w/c equal to 0.45 in RAC combined with w/c for RCA equal to 0.65).

Since it was not possible to analyse the variance (replication number in this series was equal to three), the results in this experiment should rather be interpreted as an indication at a lack of worsening in water absorption due to biomodification.

The analyses carried out prove that the biomodification is an effective means to improve sorption in RCA of better quality (w/c ratio equal to 0.45 – full diagram – 0 to 72h), despite the fact that during the first hour of measurements (lower diagram) concrete samples from unmodified RCA_45 (obtained from parent concrete with w/c equal to 0.45) presented the lowest sorption among all series. However, the difference was not particularly significant in comparison to sorption of concrete samples from biomodified RCA_45 (see Fig. 2).

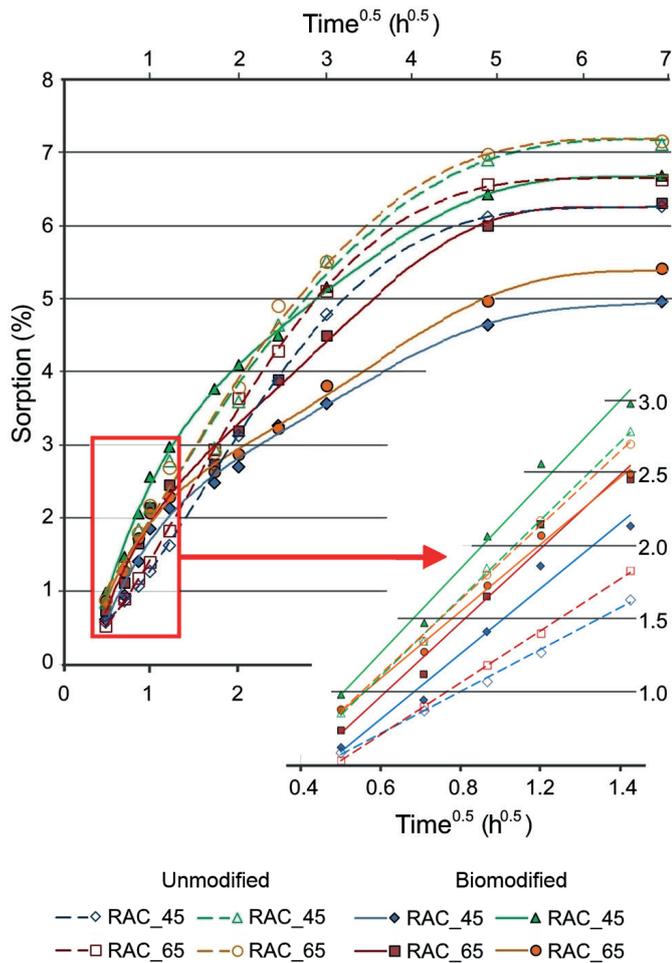


Fig. 2. Variation in time of sorption for concretes from RCA, unmodified and subjected to biodeposition

It was proven for these series by the variance analysis (Fig. 3), which was possible to carry out due to the normal type of distribution for sorption as the variable (Fig. 4). On the other hand, no statistical significance was found in the comparison of pairs – reference concrete (from unmodified aggregate) and biomodified concrete (from aggregate subjected to biodeposition) in the case of w/c ratio equal to 0.65. Lowering the sorption of concrete does not interfere with the observed deterioration of the consistency of the concrete mix. It may be associated with a reduction in the diameter of capillary pores, and related to higher capillarity in the mortar area of recycled grains. The

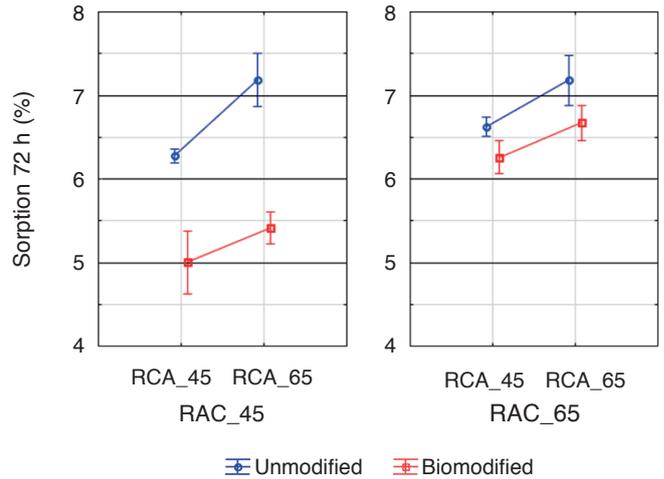


Fig. 3. Sorption of concretes from RCA after 72 h (S_{72h})

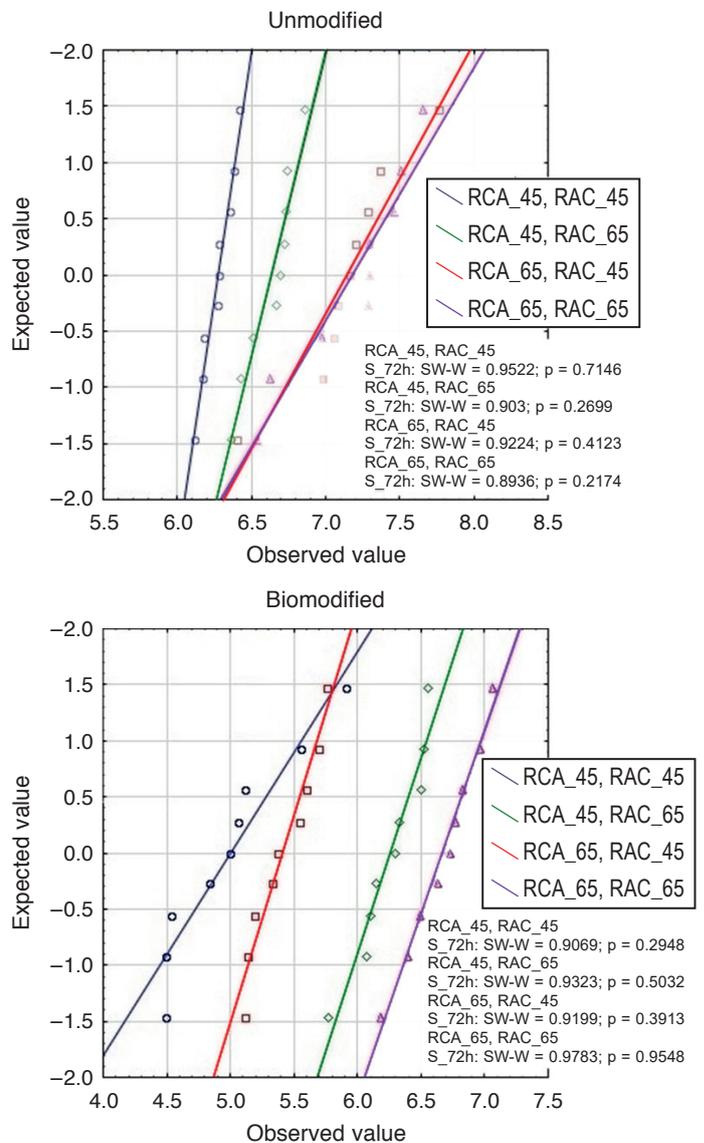


Fig. 4. Results of Shapiro-Wilk test. Note: S_{72h} – sorption after 72 h; SW-W – Shapiro-Wilk test; p – p-value

higher capillarity of the aggregate contributes to the suction of water and thus to the local (in the area of interfacial transition zone (ITZ) of RCA-cement paste) lowering water to cement ratio of concrete. Moreover, in many studies [53–57] a positive effect on the microstructure of ITZ in presence of calcite was found. The latest research [58] indicates that the beneficial effect of biomineralisation is manifested by the sealing of ITZ structure. This phenomenon may explain the reduction in sorption observed in our studies.

4. Conclusions

The following conclusions can be formulated based on the tests carried out and the analysis of their results, supported by the data from literature:

- A significant influence of biomodification of recycled concrete aggregate was found in the simplified testing of concrete sorption. In the case of recycled aggregate concrete with w/c ratio equal to 0.45, a decrease of sorption was equal to 20–25% for concretes with aggregate developed from parent concretes with w/c ratio = 0.45 and 0.65, respectively.
- Influence of biomodification on concrete strength turned out to be statistically insignificant independently of the quality of recycled concrete aggregate (in diversification of parent concretes).
- Differences in water absorption of concretes were small, but with some advantage in the case of the biomodified aggregate.
- Biomodification was more effective in the case of recycled aggregate concretes based on aggregate developed from parent concretes of better quality, as indicated by a lower value of w/c ratio.

The results of experiments are not uniquely interpretable. They do not indicate clearly at any positive influence of biomodification, as is shown in many papers, especially devoted to cement matrix composites made from traditional aggregate. The different character of recycled concrete aggregate when compared to the traditional aggregate generally does not allow for obtaining spectacular effects by use of biodeposition method. However, the number of performed experiments is still too small and the research should be extended, first of all, the method of application of microbiological material on cement matrix composited has to be diversified. One should also take care of qualitative and quantitative choice of calcium salts. Expansion of this research does not guarantee any success and it is possible that there will be no possible optimization in the biomodification of recycled concrete aggregate and the resulting concrete types. Still, the increased number of experimentations carried out in a rational way, with a scientific hypothesis, gives a chance to enhance knowledge on application of biodeposition methods in the technology of recycled aggregate concrete.

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