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ALEKSANDAR RADEVIĆ[1]
IVA DESPOTOVIĆ[2]
DIMITRIJE ZAKIĆ[1]
MARKO OREŠKOVIĆ[1]
DRAGICA JEVTIĆ[1]

[1]University of Belgrade, Faculty of Civil Engineering, Belgrade, Serbia
[2]Belgrade University, College of Applied Studies in Civil Engineering and Geodesy, Belgrade, Serbia

SCIENTIFIC PAPER

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INFLUENCE OF ACID TREATMENT AND CARBONATION ON THE PROPERTIES OF RECYCLED CONCRETE AGGREGATE

Article Highlights

- Two methods to enhance coarse recycled concrete aggregate quality are used (HCl and CO_2 treatment)
- Carbonation process has positive effects on improving physical and mechanical properties of RCA
- HCl treatment caused the increase in chloride content and the reduction of sulfate content in RCA
- If RCA is obtained from high-quality concrete, quality improving procedures are not necessary

Abstract

Recycled concrete aggregate (RCA), obtained by crushing of original (old) concrete, consists of natural aggregate grains and a cement mortar matrix. The presence of old adhered cement mortar, which has higher porosity than natural aggregate, causes unfavourable properties of RCA. The research conducted in order to improve the quality of RCA and to enable its greater application in the construction industry is presented in this paper. Therefore, RCA was subjected to quality improvement treatments with hydrochloric acid and carbon dioxide (accelerated carbonation). The first procedure was aimed at partially removing the adhered cement mortar and the second at reinforcing the cement matrix. The physical, mechanical and chemical properties of all three types of RCA were tested. After the pre-soaking acid treatment (0.1 mol/dm^3 HCl), RCA showed reduced water absorption (up to 3%); the process of accelerated carbonation also led to reduced water absorption (13-20%) as well as to improved mechanical properties (~10%). A scanning electron microscopy investigation revealed that the carbonation process, as expected, significantly reduces porosity of RCA. The overall results show that if RCA is obtained by crushing of compact, high-quality concrete, the procedures of aggregate quality improvement are not necessary.

Keywords: accelerated carbonation, pre-soaking in acid, recycled concrete aggregate.

The lack of good-quality natural aggregate in urban areas and increased distance between the sources of natural aggregate and construction sites, as well as the problem of removal and disposal of large quantities of demolished concrete waste, represent significant problems in contemporary civil engineering. Natural coarse aggregate (particle size > 4 mm) constitutes a major part of concrete and, consequently, the annual global demand for construction aggregate today exceeds 26 billion t [1]. A possible solution for the aforementioned problems could be recycling of construction and demolition (C&D) waste materials, primarily concrete. In the Republic of Serbia, appropriate mechanisms and regulations for the management of industrial and construction waste,

Correspondence: A. Radević, University of Belgrade, Faculty of Civil Engineering, Bulevar kralja Aleksandra 73, 11000 Belgrade, Serbia.
E-mail: aradevic@grf.bg.ac.rs
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based on numerous studies of waste materials, were introduced [2].

Recycled concrete aggregate (RCA), obtained by crushing of original concrete, consists of natural aggregate grains and cement mortar matrix. So far, RCA has been used for the production of new concrete and soil stabilization, as well as for materials for road pavement construction, primarily for unbound base and sub-base layers [3,4]. The presence of old cement mortar, which has higher porosity than natural aggregate, causes unfavourable properties of RCA: lower strength, higher water absorption and lower density [5,6], as well as a higher Los Angeles (LA) coefficient and fine particles content [7]. The strength of the original concrete, the crushing process and the particle size of RCA have a significant impact on the quantity of old adhered mortar, which may reach 20–-70% by weight of RCA [8,9]. This is important, since the quality and the amount of adhered mortar are the main factors influencing the physical and mechanical properties of RCA. The tiny cracks that appear during the crushing process and weak adhesion between adhered mortar and aggregate are also very important factors [10].

Therefore, extensive research has been carried out worldwide with the aim of improving recycling technologies and obtaining RCA with properties similar to natural aggregate. Removing [5,11-18] and strengthening [19–32] the adhered mortar are two common methods for improving RCA properties. The most common method for the removal of adhered mortar is pre-soaking in acid [33–35]. The usual types of applied acids are hydrochloric acid (HCl), sulfuric acid (H_2SO_4) and phosphoric acid (H_3PO_4). Since all the reaction products with HCl can be dissolved in water, some reaction products with H_2SO_4 tend to crystallize and the products with H_3PO_4 are unstable, HCl was chosen as the most effective acid [5,8,32]. The amount of adhered mortar loss is greatly influenced by the molarity of the acid [28]. Tam *et al.* investigated RCA soaking in three different strong acid solutions: HCl, H_2SO_4 and H_3PO_4 at a low concentration of 0.1 mol/dm^3 at 20 °C for 24 h [5]. The obtained results indicated that there was a considerable decrease in water absorption, improved mechanical properties and there was no adverse influence from chloride and sulfate ions on RCA. In all the aforementioned papers [5–35] only coarse RCA was investigated.

Carbonation increases the density and reduces both the water absorption and the crushing value of RCA [36,37]. Carbonation treatment of RCA improves the original interface transition zone (ITZ) of RCA, as well as the newly formed ITZ in the RCA mortar. When using the carbonation process to treat RCA, CO_2 can be stored, which reduces the global greenhouse effect [36]. The experimental results confirmed that the CO_2 curing process can densify the mortar adhered to RCA. Owing to the large specific surface area, RCA with smaller particles is more easily carbonated [37]. It was also noted that during the carbonation process, the pH value of RCA inevitably decreases [38]. During the cement hydration process, $Ca(OH)_2$ reacts with CO_2 to form $CaCO_3$, which is mainly responsible for carbonation. Apart from these components, the presence of other hydration products like calcium silicate hydrate (C-S-H), calcium hydroxide (C-H) and ettringite, highly influences the chemical process of carbonation [39].

The reaction product $CaCO_3$ precipitates in the pore space of the system and densifies the whole microstructure. According to reactions (1) and (2), after carbonation the solid volume may be increased by 11.8% based on reaction (1) and about 23% based on reaction (2) [40]:

$$\mathrm{Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O} \quad (1)$$

$$\mathrm{C-S-H + CO_2 \rightarrow CaCO_3 + SiO_2 \cdot}\, n\mathrm{H_2O} \quad (2)$$

The objective of this research was the investigation of the influence of different quality improvement treatments on the properties of RCA. Two treatments were applied: accelerated carbonation and pre-soaking in HCl. The physical, mechanical and chemical properties of untreated and treated RCA were compared and the results were analysed.

EXPERIMENTAL

The RCA used in the experiment was obtained from original concrete which served as a base structure for tram tracks; hence, it was not subjected to harmful conditions. The results obtained by testing of core specimens, showed that this concrete had a compressive strength class of C35/45 according to EN 206:2013. The original concrete was crushed in order to produce RCA with a maximum aggregate size of 22.4 mm. Regarding its composition, the RCA consisted of original concrete (98%), asphalt (1.2%) and brick debris (0.8%).

The RCA, obtained in the aforementioned manner, was classified into four fractions: 0/4, 4/8, 8/16 and 16/22.4 mm, using the standard sieving method (according to EN 933-1:2012). Numerous studies have shown that the use of fine RCA (0/4 mm) is not recommended for concrete production, due to the large specific surface area of grains, which requires an increased amount of water [1,13,24,41]. Also, it is estimated that more than 80% of concrete produced in Serbia today is made using aggregates with a nominal grain size of 16 mm. Consequently, it was decided to carry out tests only on coarse RCA (fractions 4/8 and 8/16 mm) whose particle size distribution is shown in Figure 1.

The chemical composition (major and minor chemical elements) of a representative sample of RCA, obtained by mass spectrometry, is shown in Table 1. Measurements were conducted on both macrostructural elements of RCA: natural aggregate and adhered cement mortar. These results show that the natural aggregate used for production of the original concrete consisted mainly of silicate-based (SiO_2) grains.

In order to improve the quality of the recycled aggregate, which would enable its greater application in the construction industry, RCA was subjected to treatments with HCl and CO_2. The first procedure was aimed at partially removing the adhered cement mortar and the second at reinforcing the cement matrix.

During the pre-soaking treatment with HCl, RCA was immersed in a 0.1 mol/dm^3 solution for 24 h, in accordance with previous investigations [13]. After this procedure, the aggregate was washed in distilled water and oven-dried at 100 °C. Another sample of RCA was exposed to the process of accelerated carbonation in a Memmert ICH 260 C chamber, at constant conditions: CO_2 content was 4%, temperature 20 °C and relative air humidity 55% (in accordance with the pre-standard prCEN/TS 12390-12). During the whole process, the change in mass of the RCA samples was measured every 72 h. The accelerated carbonation of RCA was performed until mass stabilization (reached after 21 days of treatment) [42]. The period of laboratory testing approximately corresponded to one year of carbonation in natural conditions in urban areas (CO_2 concentration up to 0.3% [43]). This was calculated using the following relation [44,45]:

$$t_{NCT} = \frac{[CO_2]_{ACT}}{[CO_2]_{NCT}} t_{ACT} = 21\frac{4}{0.3} = 280 \text{ days} \approx 1 \text{ year} \quad (3)$$

where t_{ACT} is the exposure time during the accelerated test [days], t_{NCT} is the exposure time for specimens subjected to a natural concentration of CO_2 [days], $[CO_2]_{ACT}$ is the concentration of CO_2 during the accelerated test [%], and $[CO_2]_{NCT}$ is the concentration of CO_2 under natural conditions (%).

On all aggregate samples – untreated RCA, RCA treated with HCl ($RCA_{(HCl)}$) and carbonated RCA ($RCA_{(CO_2)}$), the following aggregate properties were tested: apparent particle density, as well as the density of saturated and oven-dried aggregate (EN 1097-6:2007), water absorption (EN 1097-6:2013), resistance to fragmentation by the Los Angeles method (EN 1097-2:2013), changes in weight after treatment, pH value (EN 16192:2011/EN 12457 (1-4):2002), sulfate and chloride concentration (EN 16192/EPA

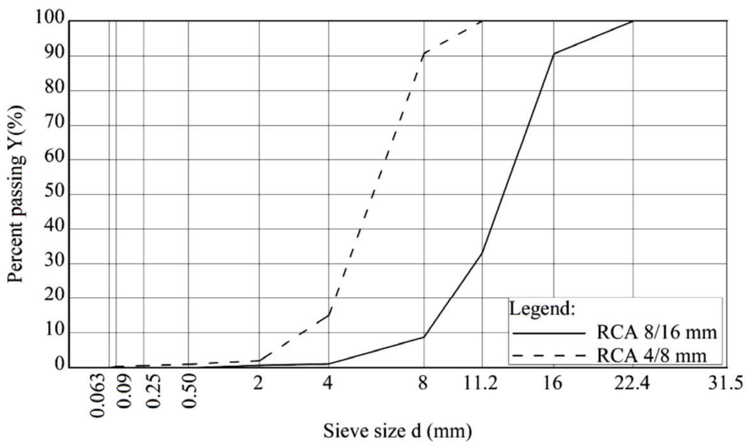

Figure 1. Particle size distribution of RCA.

Table 1. Chemical composition of a representative sample of RCA

Location	Composition, %						
	O	Mg	Al	Si	K	Ca	Fe
Natural aggregate	67.64	-	-	31.88	-	0.48	-
Adhered cement mortar	67.88	0.40	0.72	2.17	0.21	28.25	0.37

9056:2007) and microstructure, using scanning electron microscopy (SEM).

RESULTS AND DISCUSSION

For the purposes of this research, the physical and mechanical properties of all three types of RCA (untreated, acid-treated, and carbonated) were determined. The results are shown in Table 2.

Regarding the physical properties of RCA, the results obtained after HCl treatment showed that water absorption was reduced by 3.33 and 2.50%, for fractions 4/8 and 8/16 mm, respectively. At the same time, the density of RCA was higher compared with the untreated aggregate; for example, ρ_{rd} increased by 1.65% for fraction 4/8 mm and by 0.80% for fraction 8/16 mm. Thereby, the mass loss of the aggregate after HCl treatment, was 4.30 (for fraction 4/8 mm) and 2.90% (for fraction 8/16 mm) which corresponded with the results from study [46]. An inverse dependence was observed between water absorption and aggregate density, as a result of the partial removal of the porous cement mortar. This was also confirmed by other researchers [8,13,25,36,37].

Significantly better results were achieved after the carbonation process: a decrease in water absorption by 13.3 (fraction 4/8 mm) and 20% (fraction 8/16 mm), which is in accordance with results obtained by [40]. Also, the increase in the oven-dried density in the amount of 1.40 (fraction 4/8 mm) and 0.40% (fraction 8/16 mm) was discovered. After the carbonation process was completed, the total aggregate mass was increased by 0.90 (fraction 4/8 mm) and 1.40% (fraction 8/16 mm). These properties of RCA are the result of the formation of carbonation products in the micro-cracks of the cement mortar.

In Figure 2 the correlation between water absorption and RCA density, depending on the applied treatment, is shown. It can be concluded that the larger aggregate fraction is characterized by higher water absorption, regardless of the applied treatments. Trends of water absorption reduction are similar for both fractions.

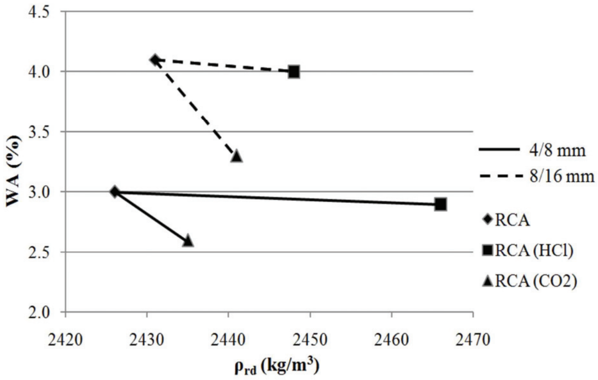

Figure 2. Trends of water absorption change as a function of RCA density.

Results of the mechanical properties testing of RCA, based on the measurement of resistance to fragmentation, show that all three types of RCA have a satisfactory quality (Los Angeles coefficient lower or approximately equal to 30%). At the same time, there was no significant difference between the untreated and acid-treated aggregate, which indicates a very good relationship (adhesion) between the grains of the natural aggregate and the old cement mortar. In the case of carbonated RCA, there was an increase in the aggregate resistance to fragmentation, amounting to approximately 10 %, compared with untreated RCA.

The chemical properties of RCA, before and after the treatment, were determined. The results are shown in Table 3.

The concentrations of sulfates and chlorides, as potentially harmful compounds that can cause deterioration of concrete, *i.e.*, corrosion of steel reinforcement, was also controlled. The results showed that HCl treatment significantly increased the concentration of chlorides in the aggregate, by 20.9% for

Table 2. Physical and mechanical properties of untreated and treated RCA; ρ_a - apparent particle density, ρ_{ssd} - saturated and surface-dried particle density, ρ_{rd} - oven-dried particle density, WA - water absorption, LA - Los Angeles coefficient, Δm_1 - percentage of change in mass after treatment (acid or carbonation); Δm_2 - percentage of change in mass after treatment and sieving

Parameter	RCA		$RCA_{(HCl)}$		$RCA_{(CO_2)}$	
	4/8 mm	8/16 mm	4/8 mm	8/16 mm	4/8 mm	8/16 mm
ρ_a / kg m^{-3}	2618	2699	2681	2654	2667	2657
ρ_{ssd} / kg m^{-3}	2499	2530	2547	2525	2522	2523
ρ_{rd} / kg m^{-3}	2426	2431	2466	2448	2435	2441
WA / %	3.0	4.1	2.9	4.0	2.6	3.3
LA / %	31.5		32.0		28.4	
Δm_1 / %	-	-	-2.3	-2.2	0.9	1.4
Δm_2 / %	-	-	-4.3	-2.9	0.9	1.4

Table 3. Chemical properties of untreated and treated RCA

Parameter	RCA		$RCA_{(HCl)}$		$RCA_{(CO_2)}$	
	4/8 mm	8/16 mm	4/8 mm	8/16 mm	4/8 mm	8/16 mm
pH value	10.7	11.8	10.6	11.5	9.5	9.8
Concentration of SO_4^{2-} (mg/kg)	1212	605	514	330	1008	576
Concentration of Cl^- (mg/kg)	177	242	214	533	175	271

fraction 4/8 mm and by 120.2% for fraction 8/16 mm, which was expected. At the same time, the concentration of sulfates significantly decreased after this treatment (about 50%), which can be explained by the dissolution and removal of the old cement mortar; this was also confirmed by other researchers [5]. On the other hand, the carbonation process practically had no effect on the change of concentrations of chlorides or sulfates in RCA. As expected, during the HCl treatment the pH value did not change significantly, while after the carbonation process, it decreased from 10.7 to 9.5 for fraction 4/8 mm and from 11.8 to 9.8 for fraction 8/16 mm.

The microstructure of untreated and treated RCA was analysed using SEM, model JEOL JSM-6610LV. The SEM images were taken by changing the magnification from 5 to 100 µm. The accelerating voltage was 20 kV. Characteristic SEM images are shown in Figures 3–5.

The morphology of untreated RCA was rough and irregular with a highly porous structure where adhered mortar was widely spread at different thicknesses causing surface heterogeneity. SEM images clearly indicate the presence of many various voids and particles without specific shape and size. The remains of acid-eroded old cement mortar with numerous voids of irregular shape, whose length reaches up to 20 µm are shown in Figure 4. On the other hand, the carbonation process has significantly reduced the porosity of RCA (pore sizes up to 2 µm, see Figure 5), which is in accordance with other obtained results related to physical and mechanical properties.

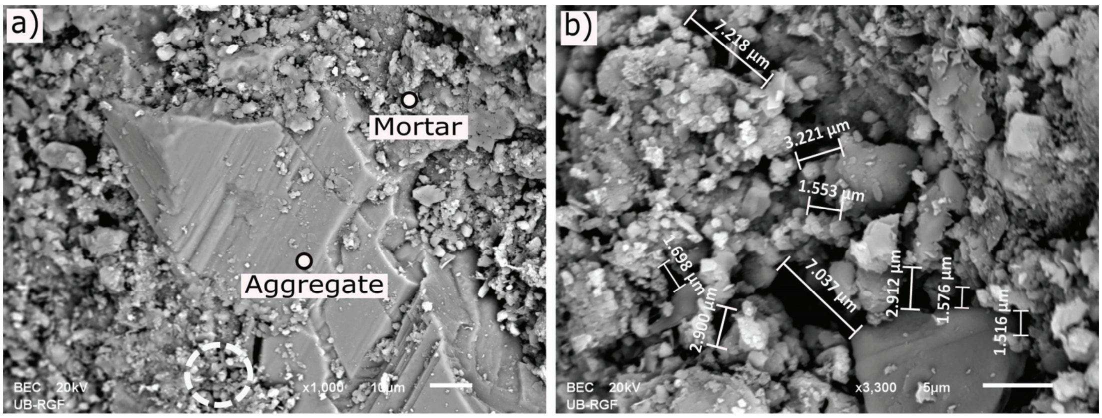

Figure 3. a) Surface microstructure views of RCA grain without treatment and b) magnified detail.

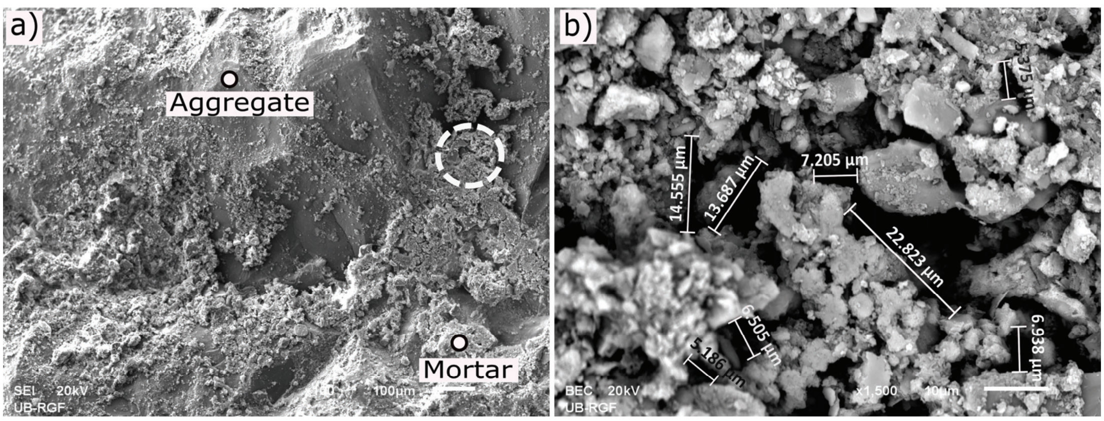

Figure 4. a) Surface microstructure views of RCA grain after HCl treatment and b) magnified detail.

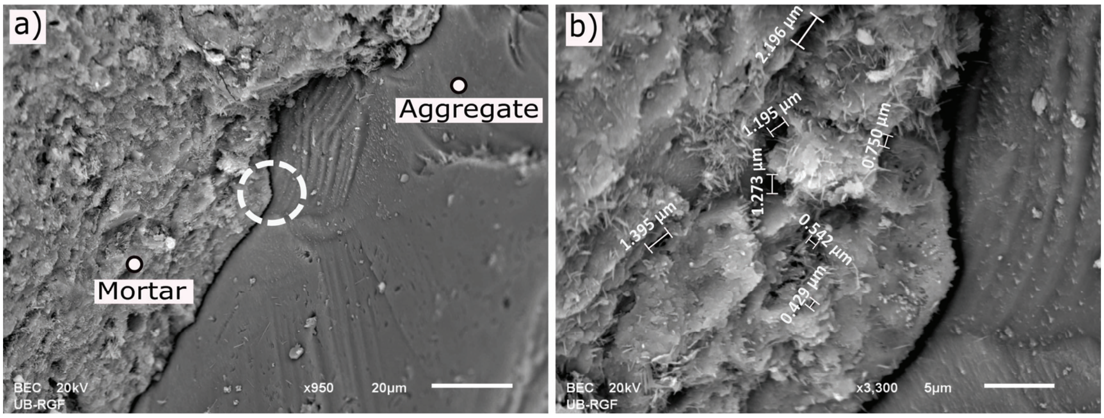

Figure 5. a) Surface microstructure views of RCA grain after CO_2 treatment and b) magnified detail.

CONCLUSIONS

In the study, the physical, mechanical and chemical properties of three types of coarse RCA were tested: untreated RCA, RCA subjected to a pre-soaking treatment in HCl and RCA subjected to accelerated carbonation. The first treatment was aimed at partially removing adhered cement mortar and the second at reinforcing the cement matrix. After the acid treatment with HCl (0.1 mol/dm^3), coarse RCA showed reduced water absorption by 2-3% (compared with untreated RCA), while its density was increased by 1%. At the same time, mechanical properties practically did not change. A chemical analysis showed that HCl treatment caused an increase of chloride concentration and a reduction of sulfate concentration, while the pH value remained the same. SEM images of aggregate after acid treatment clearly indicated the presence of many various voids with different sizes (5-23 μm), which were approximately three times higher compared with untreated RCA.

After the process of accelerated carbonation, better results were achieved, in terms of reducing water absorption (13-20% compared with untreated RCA) and improving mechanical properties (~10%), while density was increased by less than 1%. As expected, accelerated carbonation led to a decrease of the pH value, while it did not affect the chloride and sulfate concentrations in the aggregate. The microscopic analysis performed by SEM confirmed the obtained results of the experimental study. SEM images showed that voids in RCA after carbonation did not exceed 2 μm.

The correlation between water absorption and RCA density showed that the larger aggregate fraction (8/16 mm) was characterized by higher water absorption than the smaller fraction (4/8 mm), regardless of the applied treatment. The established trend lines of water absorption reduction were similar for both RCA fractions.

It should be noted that the coarse RCA used in this study was obtained by crushing of relatively high-quality concrete (compressive strength class C35/45), which was also verified by water absorption and Los Angeles coefficient values. It has been found that when using this type of RCA, quality improvement procedures are not necessary. Therefore, such RCA can be successfully used for making cement or asphalt concrete, without any prior treatment.

As for the ecological contribution, the aim of the study was to improve the quality of RCA and to enable its greater application in the construction industry. In Serbia, as well as in other European countries, there is a growing pressure to increase the use of alternative materials in construction applications. Utilization of RCA may have many ecological benefits, such as saving natural resources, reducing the demand for greater landfill capacities, storing CO_2 and decreasing its emission, as well as having an aesthetic influence on the environment.

The future investigation will be extended with research on newly prepared concrete, based on untreated and treated RCA. This research will also include testing of Hg porosimetry and XRD analysis of three types of RCA. In that manner, the research shall be complemented with the results indicating possible influence of RCA on the mechanical, mineralogical and textural properties of the new concrete.

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ALEKSANDAR RADEVIĆ[1]
IVA DESPOTOVIĆ[2]
DIMITRIJE ZAKIĆ[1]
MARKO OREŠKOVIĆ[1]
DRAGICA JEVTIĆ[1]

[1]Univerzitet u Beogradu, Građevinski fakultet, Bulevar kralja Aleksandra 73, 11000 Beograd, Srbija
[2]Visoka građevinsko-geodetska škola Beograd, Hajduk Stankova 2, 11000 Beograd, Srbija

NAUČNI RAD

UTICAJ DELOVANJA KISELINE I KARBONATIZACIJE NA SVOJSTVA AGREGATA OD RECIKLIRANOG BETONA

Agregat od recikliranog betona (RCA), dobijen drobljenjem originalnog (starog) betona, sastoji se od zrna prirodnog agregata i cementne matrice. Prisustvo zaostalog cementnog maltera, koji ima veću poroznost od prirodnog agregata, prouzrokuje nepovoljna svojstva RCA. U ovom radu je prikazano istraživanje koje je obavljeno u cilju poboljšanja kvaliteta RCA i omogućavanja njegove šire primene u građevinskoj industriji. U tom smislu, RCA je izložen tretmanima za poboljšanje kvaliteta na bazi hlorovodonične kiseline i ugljendioksida (ubrzana karbonatizacija). Prvi tretman je imao za cilj uklanjanje zaostalog cementnog maltera, a drugi ojačanje cementne matrice. Ispitivana su fizička, mehanička i hemijska svojstva sva tri tipa RCA (tretiranih i netretiranih). Nakon prethodnog potapanja u kiselinu (0,1 mol/dm^3 HCl), RCA je pokazao smanjeno upijanje vode (do 3%); postupak ubrzane karbonatizacije je takođe doveo do smanjenja upijanja vode (13-20%), kao i do poboljšanja mehaničkih svojstava (~10%). SEM (Scanning Electron Microscopy) analiza je pokazala da proces karbonatizacije, kao što je bilo očekivano, značajno smanjuje poroznost RCA. Generalno, rezultati ispitivanja pokazuju da, u slučaju RCA dobijenog drobljenjem kompaktnog betona visokog kvaliteta, nije neophodno sprovođenje tretmana za poboljšanje kvaliteta agregata.

Ključne reči: ubrzana karbonatizacija, prethodno potapanje u kiselinu, agregat od recikliranog betona.