Paper published in:

A. Cuccurullo, Y. Hou, P.-Y. Mahieux, C. Perlot, A. Aït-Mokhtar, J. Lux, P. Turcry, D. Gallipoli

(2025).

Effect of relative humidity and carbonation on the mechanical behavior of compacted fine recycled aggregates.

Materials and Structures, 58: 248

https://doi.org/10.1617/s11527-025-02748-0

Effect of relative humidity and carbonation on the mechanical behavior of compacted fine recycled aggregates

Alessia Cuccurullo[1], Yunlu Hou[2], Pierre-Yvex Mahieux[3], Celine Perlot[4,5], Abdelkarim Aït-Mokhtar[3], Jérôme Lux[3], Philippe Turcry[3], Domenico Gallipoli[6]

[1]Laboratoire LGM, BATir Department, Université libre de Bruxelles, Belgium
[2]Institute of Rock and Soil Mechanics, Chinese Academy of Sciences, China
[3]LaSIE UMR CNRS 7356, La Rochelle Université, France
[4]Laboratoire SIAME, Université de Pau et des Pays de l'Adour, France
[5]Institut Universitaire de France, France
[6]Dipartimento di Ingegneria Civile, Chimica e Ambientale, Università degli Studi di Genova, Italy

Abstract: Recycled aggregates are primarily composed of concrete, natural stones, and bricks obtained through sorting, crushing, and sieving of construction and demolition waste. They offer a promising route toward reducing the consumption of natural resources and minimizing landfill use in civil engineering, thus supporting circular economy and contributing to more sustainable construction practices. This study specifically investigates the mechanical behavior of compacted fine recycled aggregates for road pavement base layers, with a focus on the effects of relative humidity and carbonation. Cylindrical samples with a compactness of 0.69 were subjected to controlled curing conditions over 30 and 100 days. Three relative humidity levels (53%, 65%, and 95%) and two carbon dioxide concentrations (0.05% representing natural conditions and 3% for accelerated carbonation) were also investigated. Additional samples were oven-dried to establish baseline mechanical properties under zero moisture content. All specimens were tested in a triaxial apparatus under confining pressures of 0, 100, 250, and 400 kPa to assess their shear strength and deformation behavior. The results indicate that higher CO_2 concentrations lead to a reduction in strength, contrary to expectations. Additionally, increased relative humidity correlates with lower peak strength and higher ductility. These findings underscore the critical influence of environmental curing conditions on the long-term performance of recycled aggregates in pavement applications.

Keywords: Recycled aggregates, Carbonation, Triaxial testing, Shear strength, Stiffness, Suction, Residual hydraulic activity.

List of notations

C_r Compactness

C Intercept of the failure envelope

c' Cohesion

$Ca(OH)_2$ Portlandite

CO_2 Carbon dioxide

$C-S-H$ Calcium silicate hydrate

C_2S Dicalcium silicate

C_3S Tricalcium silicate

E Apparent Young's Modulus

ε Axial strain

ITZ Interfacial transition zone

M Slope of the failure envelope

M_w Molar mass of water

n Porosity

p Mean stress

ρ_b Bulk density

ρ_d Dry density

ρ_w Unit weight of water

q Deviator stress

σ Axial stress

σ_c Confining pressure

R Constant of perfect gas

RH Relative humidity

s Suction

S_r Degree of saturation

T	Absolute temperature
φ'	Friction angle
V_s	Solids volume
V_t	Total sample volume
w	Water content

1 Introduction

Construction and demolition waste (CDW) is one of the largest industrial byproducts globally, with approximately 3 billion tons generated annually [1, 2]. In France, the construction industry alone produces around 230 million tons of inert CDW each year, while consuming nearly 400 million tons of natural aggregates [3]. This dual challenge—excessive waste generation and intensive consumption of natural resources—has led to an urgent need for more sustainable construction practices. Among these, the conversion of CDW into recycled aggregates (RA) has emerged as a promising solution. RA not only reduces pressure on landfills but also lessens dependence on natural aggregates. Ongoing research highlights their potential as versatile construction materials, particularly for road pavement base and sub-base layers, where they have shown promising mechanical properties and residual hydraulic reactivity [4,5,6,7,8,9].

Despite increasing interest, the long-term performance of RA under field-like environmental conditions remains insufficiently understood. Recycled aggregates are heterogeneous composites, typically comprising natural aggregates bonded with old cement paste, along with smaller proportions of materials such as bitumen, ceramic brick, glass, wood, and plastic. This variability significantly influences their engineering behavior, making accurate sorting and characterization essential for consistent performance [10,11,12,13,14,15,16]. A phenomenon unique to RA is “re-cementation” [17], which refers to renewed binding due to hydration of unreacted cement or pozzolanic reactions with fine constituents. For instance, Poon et al. [18] observed that residual anhydrous cement in fine recycled concrete aggregates contributes to self-cementing behavior in pavement sub-base materials. Vegas et al. [19] further

demonstrated that the presence of ceramic particles induces pozzolanic reactions that enhance the bearing capacity of compacted RA.

Field and laboratory investigations have confirmed that re-cementation can significantly improve the mechanical properties of RA over time. Chai et al. [20] reported a progressive increase in stiffness in pavement layers containing RA in both California and South Africa, attributed to re-cementation under service conditions. This time-dependent behavior was further linked to factors such as traffic loading and material depth. Paige-Green [21] observed newly formed cementitious products in pavement cores incorporating RA, while Jitsangiam et al. [22] found continued hydration of cement residues in laboratory-cured samples. These studies underscore the role of re-cementation in enhancing long-term mechanical performance but also suggest that its structural implications and potential side effects (e.g., reduced permeability, reflective cracking) require further evaluation.

In addition to hydration and pozzolanic activity, the cementitious phases in RA can react with atmospheric carbon dioxide CO2 through carbonation. This chemical process forms calcium carbonate ($CaCO_3$), which reduces porosity and improves stiffness. While many studies focus on CO_2 curing to improve RA for concrete use, recent research emphasizes its potential for carbon sequestration. Arm [23] showed that compacted RA exhibits time-dependent stiffness increases due to carbonation, in contrast to the stable behavior of natural aggregates. However, the rate and effectiveness of carbonation depend on binder composition, moisture levels, temperature, and pore structure. Although beneficial, most existing studies address short-term effects in laboratory settings, and long-term durability under real environmental conditions is not well understood.

Hou et al. [16,24,25,26] explored the combined effects of relative humidity and carbonation on the mechanical behavior of RA. Samples were cured for 21 days at 53%, 65%, and 95% relative humidity (RH) under controlled temperature of 20 ± 5 °C. Results showed that dynamic modulus increased as humidity decreased, with maximum stiffness observed at 53% RH due to enhanced carbonation and stronger inter-particle capillary bonds. Conversely, higher humidity (95%) favored continued hydration and the formation of crystalline products, resulting in gradual strength gains beyond 21 days. Compressive strength increased during early curing but plateaued with time. Interestingly, accelerated carbonation had limited effect on long-term performance, suggesting that most stabilizing reactions occur during earlier phases, such as crushing and transport.

Despite these advances, critical knowledge gaps remain regarding the long-term mechanical evolution and durability of RA under realistic environmental conditions. Specifically, the interplay between moisture content, CO_2 concentration, curing duration, and the development of hydration, pozzolanic, and carbonation reactions has not been comprehensively examined. This study directly addresses these gaps by systematically investigating how these factors influence the mechanical properties of compacted recycled aggregates derived from demolition waste. Through detailed testing of shear strength, Young's modulus, and ductility under varied environmental conditions, this work provide new insights into the long-term feasibility and reliability of RA for pavement applications. The findings contribute to both scientific understanding and the advancement of sustainable design strategies that reflect the real conditions encountered during road construction and service life.

2 Testing material and experimental program

The amount of water within compacted recycled aggregates affects both inter-particle capillary bonds and chemical activity of cementitious products which have significant consequences on strength and stiffness. This aspect was experimentally investigated in the present work by testing samples of compacted fine recycled aggregates exposed to natural and accelerated carbonation at a temperature *T* of 20±5°C and three relative humidity levels RH of 53%, 65% and 95% for either 30 or 100 days. These relative humidities were chosen to favor, or not, the diffusion of CO_2 in the inter and intra-granular porosity in natural CO_2 concentration of about 0.05% according to Hou et al. [26]. Additional tests were carried out on samples which were dried in an oven at 105°C for a minimum of three days to establish a reference condition corresponding to the absence of free pore water.

Samples were manufactured and equalized at the LaSIE laboratory of La Rochelle Université (France) using the 0/6 mm particle size fraction of the recycled aggregates supplied by a waste reprocessing plant in La Rochelle. On-site, construction and demolition wastes were crushed, sorted manually then stored before testing in laboratory. About 99.2% of this fraction was made of crushed concrete, mortar and natural aggregates with particle sizes comprised between 4 mm and 6 mm, which complied with current guidelines for road construction [27-28]. The particle density and the initial water content of the material were 2.5 g/cm^3 and 3.4 %, respectively.

Dried recycled aggregates and water were mixed with a mechanical blender to make cylindrical samples with diameter and height of 50 mm and 100 mm in accordance with the European Standard NF EN 13286-53 [29] (Fig. 1). The water content of 10% and compactness $C_r = V_s/V_t = 0.68$, where V_s and V_t are the solids volume and the total sample volume, were chosen to produce samples with an unconfined compressive

strength of at least 1 MPa, which is the minimum recommended by road subgrade guidelines, e.g. NF P94-102-1 [30] and LCPC/SETRA [31]. A sample aspect ratio of 2 was chosen to limit the confining effect of friction between the sample extremities and the press plates during triaxial tests.

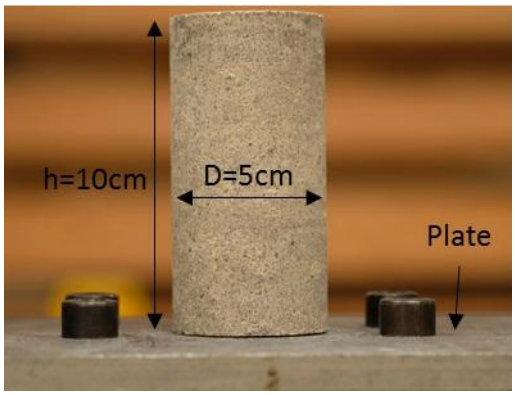


Fig.1 Cylindrical sample of compacted fine recycled aggregates

Different samples were subjected to natural and accelerated carbonation inside three enclosures with relative humidities of 53%, 65% and 95%, respectively. Accelerated carbonation was only imposed at the relative humidity of 65% and, in this case, a mechanical ventilation system was used to homogenize carbon dioxide concentration inside the enclosure. Different saturated salt solutions were introduced inside the carbonation enclosures to impose the required relative humidity levels (i.e. magnesium nitrate, ammonium nitrate and potassium nitrate for relative humidities of 53%, 65% and 95% respectively) while the ambient temperature was maintained at 20°±5C. During natural carbonation, the carbon dioxide concentration inside the enclosures was always equal to the ambient level of 0.05% while during accelerated carbonation, the carbon dioxide concentration was increased to 3%. To investigate the role of curing time, the samples were maintained in the above conditions for either 30 or 100 days. Fig. 2 shows the experimental setups for both natural and accelerated carbonation [25].

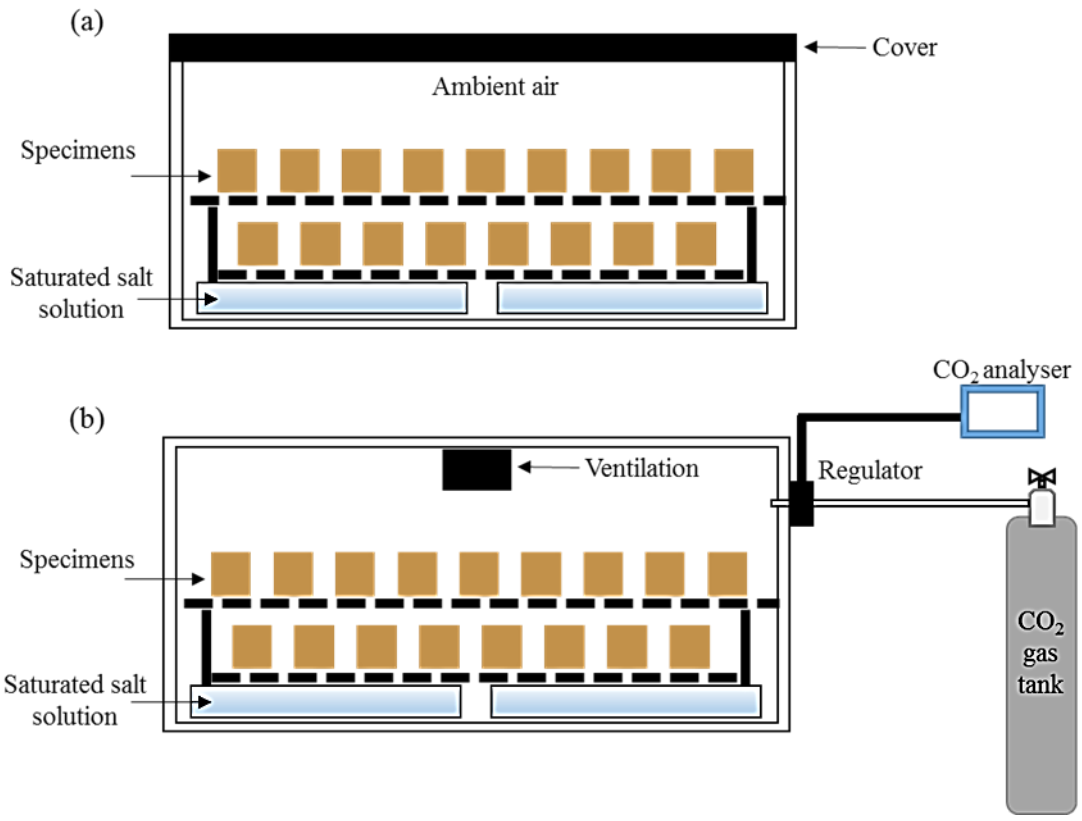


Fig. 2 Experimental carbonation setups: CO2=0.05% at 53%, 65% and 95% RH (a) and CO2=3% at 65% RH (b) [11]

The carbonated compacted samples were then sealed and shipped to the SIAME laboratory of the Université de Pau et des Pays de l' Adour (France) for triaxial testing. The interval between the end of carbonation and the triaxial tests was relatively short, averaging approximately 4-5 days. To correct any potential minor change in moisture content during shipping, the samples were re-equilibrated before testing to their target relative humidity and temperature levels inside a climatic chamber (CLIMATS Type EX2221HA) or oven-dried. The average bulk density ρ_b was measured for each set of naturally and accelerated carbonated samples, as listed in Table 1. After the triaxial tests, three fragments of about 50 grams were taken at the top, middle and bottom of each sample to determine the corresponding water contents according to the French norm NF P94-050 [32]. The three measurements were generally very similar, thus confirming the uniformity of water content across the sample. The average water

content w was then calculated for each set of samples, as listed in Table 1. Based on the above measurements of bulk density ρ_b and water content w, the values of dry density ρ_d , degree of saturation S_r and porosity n were calculated for each set of samples, as listed in Table 1.

Assuming equilibrium conditions after 100 days and a constant temperature of 20±5°C, the total suction s was calculated (Table 1) according to Kelvin's equation [33] from the imposed relative humidity RH as:

$$s = -\frac{\rho_w R T}{M_w} \ln RH \quad (1)$$

where R is the constant of perfect gases [8.3145 J/mol·K], T is the absolute temperature, M_w is the molar mass of water [0.018 kg/mol] and ρ_w is the unit weight of water [1000 kg/m^3]. Recall that the soil suction is a state variable combining the distinct effects of water capillarity, adsorption and ionic interactions on the solid skeleton. It can therefore also be regarded as a measure of the negative thermodynamic potential of pore water relative to the reference level of pure water at atmospheric pressure [34].

Table 1 Samples properties after curing

	ρ_b *[G/CM^3]*	w *[%]*	ρ_d *[G/CM^3]*	S_r *[%]*	n *[%]*	s *[MPA]*
			30 days			
RH = 53%	1.73	1.94	1.69	9.13	36.0	/
RH = 65% (nat. carb.)	1.75	3.43	1.69	16.1	36.1	/
RH = 65% (acc. carb.)	1.76	1.31	1.74	6.60	34.4	/
RH = 95%	1.75	3.31	1.70	15.6	35.9	/
			100 days			
RH = 53%	1.73	0.83	1.71	4.02	35.3	87.4
RH = 65%	1.74	1.14	1.72	5.57	35.1	59.3

(nat. carb.) RH = 65%	1.75	1.55	1.72	7.63	35.0	59.3
(acc. carb.) RH = 95%	1.75	1.67	1.73	8.26	34.9	7.1
Oven-dried						
RH = 0%	**1.68**	**0**	**1.68**	**0**	**36.6**	**/**

After curing, the cylindrical samples were wrapped into latex membranes and mounted on a triaxial cell where they were confined under four distinct confinement pressures σ_c of 0 kPa, 100 kPa, 250 KPa and 400 kPa prior to shearing at constant water content until failure. These four confining pressures were selected considering that horizontal stresses inside the sub-base layer of road pavements do not, in general, exceed 400 kPa prior to shearing at constant water content until failure. Based on preliminary tests (not shown here), an axial displacement rate of 0.001 mm/s was chosen to minimize the sensitivity of the material response to the strain rate effects [35]. The triaxial tests were conducted using a saturated triaxial cell; however, the saturation phase was deliberately omitted, and the samples were confined and sheared under unsaturated conditions. This approach was chosen to specifically investigate the influence of relative humidity and water content on the mechanical behavior of the material. The samples were axially compressed while keeping the confining pressure constant and the back-pressure line kept opened to the atmosphere for draining excess pore air pressures. Evaporation of moisture through the back-pressure line was considered negligible and the water content was therefore assumed constant.

3 Results and discussion

3.1. Effect of relative humidity and accelerated carbonation

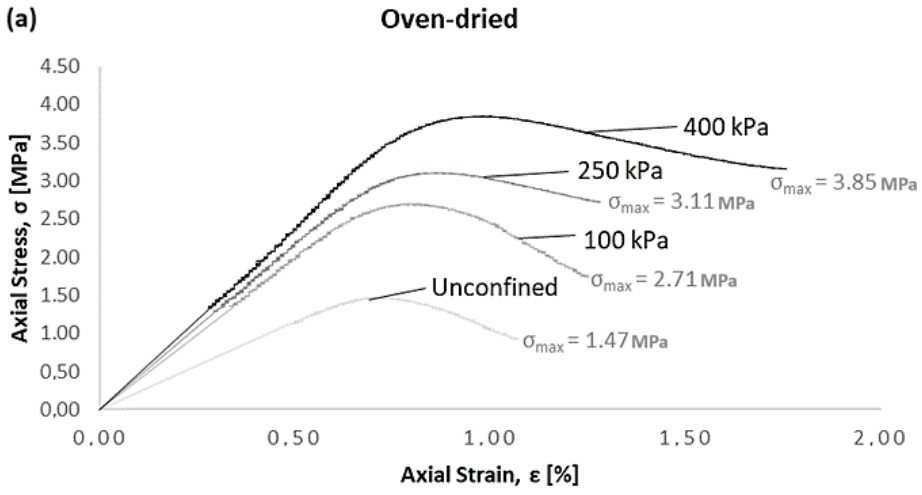
Fig. 3 (a-d) shows the stress-strain curves obtained from triaxial tests on samples which were either naturally carbonated at the three relative humidities of 53%, 65%

and 95% for 30 days or oven-dried. In all cases, the stress-strain curves are reasonably linear at low strains exhibiting, as expected, greater strength and lower post-peak softening as the confining pressure increases from 0 to 400 kPa.

The peak strength increases with decreasing relative humidity at all confining pressures, which corroborates the assumption that suction bonds particles together, thus improving the mechanical characteristics [36-37]. Interestingly, the oven-dried samples exhibit relatively high peak strength levels, which are only slightly lower than those of samples cured at the lowest relative humidity of 53%. These peak strengths are also comparable to, or even higher than, those of samples equilibrated at the intermediate and highest relative humidities of 65% and 95%, respectively. This is counterintuitive because a dry granular material is conceptually no different from a saturated one, for which the principle of effective stresses must apply. Therefore, if the samples are truly dry, no inter-granular capillary menisci should be present and, hence, strength should be lowest among all four cases. An explanation for this seemingly counterintuitive observation is that oven-dried samples are not completely dry, contrary to common assumptions. In fact, previous studies have shown that soils oven-dried at 105 °C can retain a small amount of bound water held under extremely high tension. While the adsorbed water cannot form capillary bridges, it is associated with extremely high matric suction promoting significant interparticle attraction through physicochemical forces and contributing significantly to the observed peak strength [38-40]. Drying temperatures higher than 105°C were not explored in the present study to avoid the decomposition of cement hydrate products (e.g. ettringite, calcium silicate hydrate C-S-H). Despite it is difficult to separate the effects of the three phenomena that are believed to modify strength (suction, residual hydraulic reactivity, carbonation), which are likely concurrent under natural conditions, it is reasonable to correlate the

evolution of compressive strength with the decrease in water content and the increase in capillary forces.

Hou *et al.* [25], from testing on same recycled compacted samples, showed that the mechanical strength increases during the first 30 days, reaching values 2.1 to 2.7 times higher than the initial strength. After 30 days, the strength remains almost constant. This trend was observed across all conditions and relative humidities (53%, 65%, and 95%) investigated. To evaluate the effect of capillarity, samples were saturated in water under vacuum for 24 hours, after they had been stored for 315 days at relative humidities of 53%, 65%, and 95%. Additionally, some samples were saturated immediately after compaction. The authors [25] demonstrated that capillary cohesion contributes to the mechanical strength while, over time, physical bonds between the grains have formed due to the residual chemical reactivity of the recycled aggregates. It is worth noting that after saturation, the strength of the 315-day samples was four times higher than the strength of the samples saturated at 0 days (2 MPa compared to 0.5 MPa) demonstrating that the material indeed experienced significant changes over time, independent of any capillary effects.



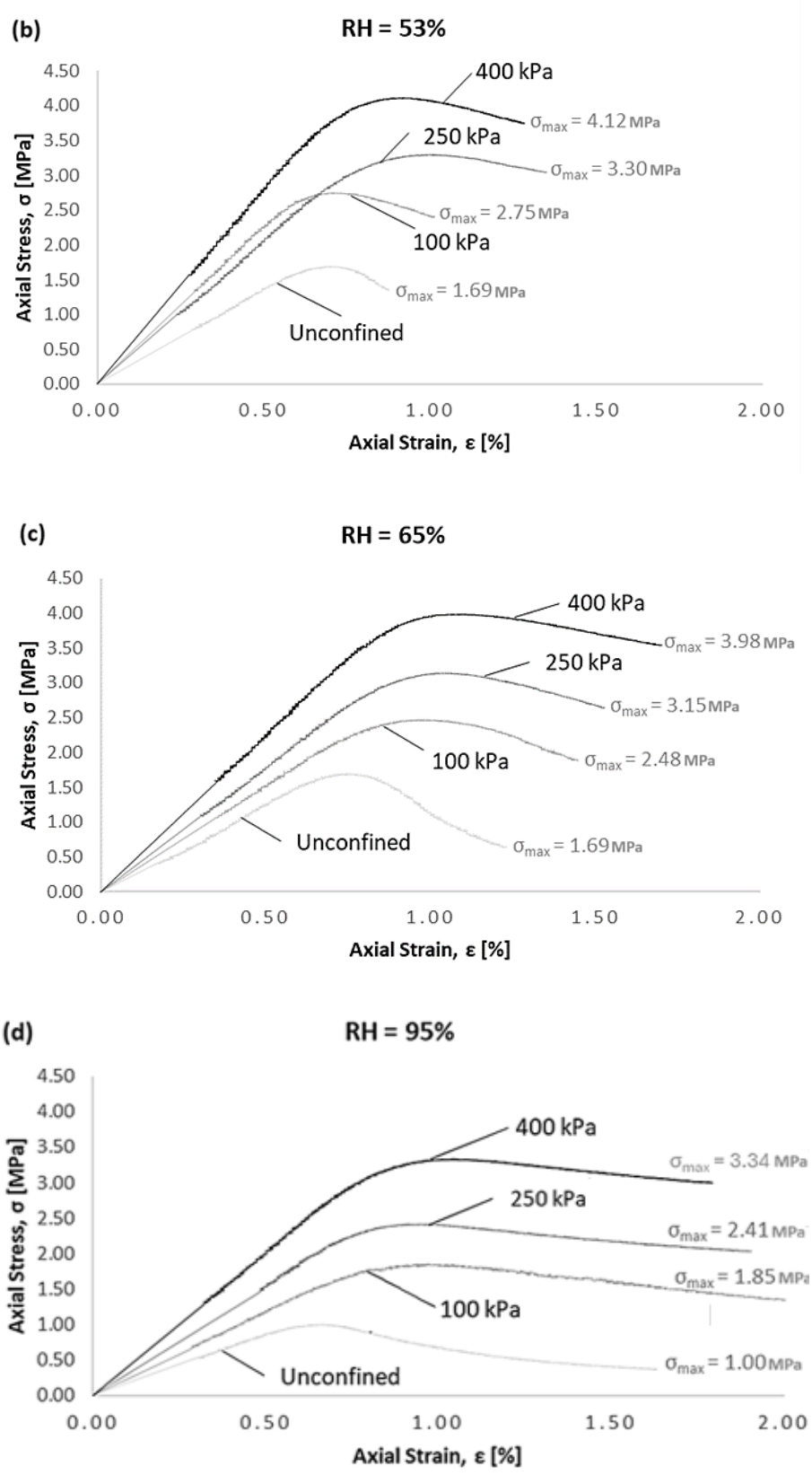


Fig. 3 Stress-strain curves obtained from triaxial tests at varying confining pressures on naturally carbonated samples: after oven-drying (a), after 30 days of curing at 53% (b), 65% (c), and 95% (d) RH

Inspection of Fig. 3 (a-d) also indicates a gentle decrease of strength after the peak with the degree of brittleness slightly increasing as the relative humidity decreases. A increasing water content therefore reduces strength while increasing the ability of the material to undergo softening before failure. Lower material brittleness corresponded to a more diffuse failure mechanism, where samples absorbed significant amounts of energy before fracturing through multiple cracks (Fig. 4). Previous studies [41] have suggested that the interfacial transition zone (ITZ), between the natural aggregate and the mortar coating, constitutes a point of weakness because of the presence of micro-cracks that have formed during previous demolition and crushing. Furthermore, other authors [42-43] have also indicated that loose porous hydrates are usually found in the ITZ of recycled aggregates, instead dense that exist in undamaged concrete. This corroborates the hypothesis of a progressive failure mechanism starting at the ITZ, where the concentration of stresses over an already weakened zone leads to the initiation and subsequent propagation of cracks. The presence of these diffused weak interfaces is coherent with the multiple fracturing pattern that has been observed at the end of triaxial shearing (Fig. 4).

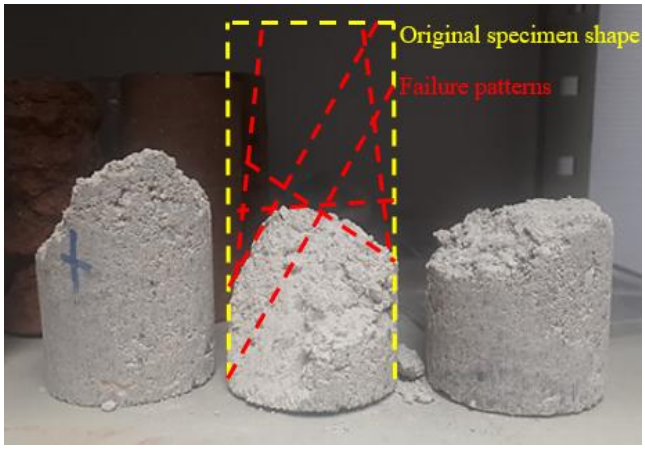


Fig. 4 Multiple fracturing failure mechanism after triaxial testing for samples equalized at 53% RH

Fig. 5 presents the stress-strain curves obtained from triaxial tests on accelerated carbonated samples at a single relative humidity of 65% for 30 days. The comparison with the corresponding curves of the naturally carbonated samples (Fig. 3c) indicates that accelerated carbonation does not increases the peak strength but rather reduces it. In accordance with previous study by Hou *et al.* [25], the strength reduction may be due to the fact that the portlandite ($Ca(OH)_2$) phase of the recycled aggregates has already been mostly carbonated during crushing at the recycling platform and subsequent transportation to the testing site. Therefore, in the absence of such reactant phase, a higher carbon dioxide concentration tends to produce the carbonation of the calcium silicate hydrate (C-S-H) gel (decalcification phenomena), thus resulting in a greater porosity and worse mechanical behavior [44-45]. A decrease in strength due to the carbonation of calcium silicate hydrate (C-S-H) gel has also been suggested in previous studies on concrete and mortar [46-49].

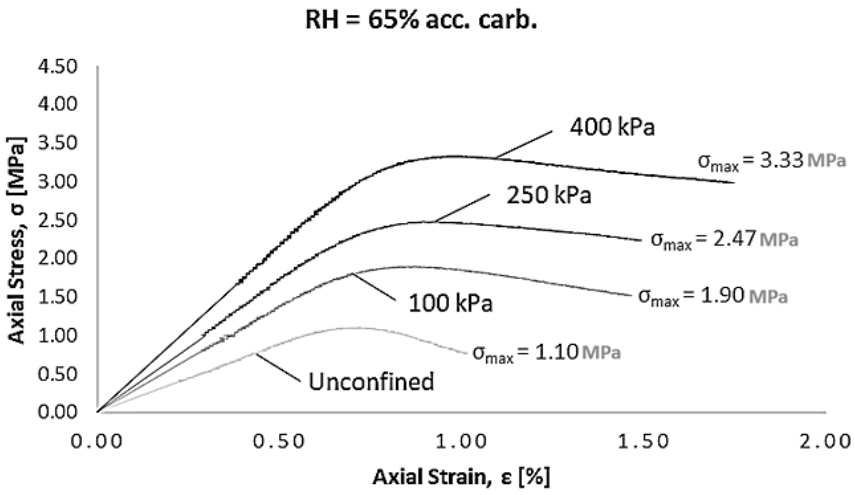
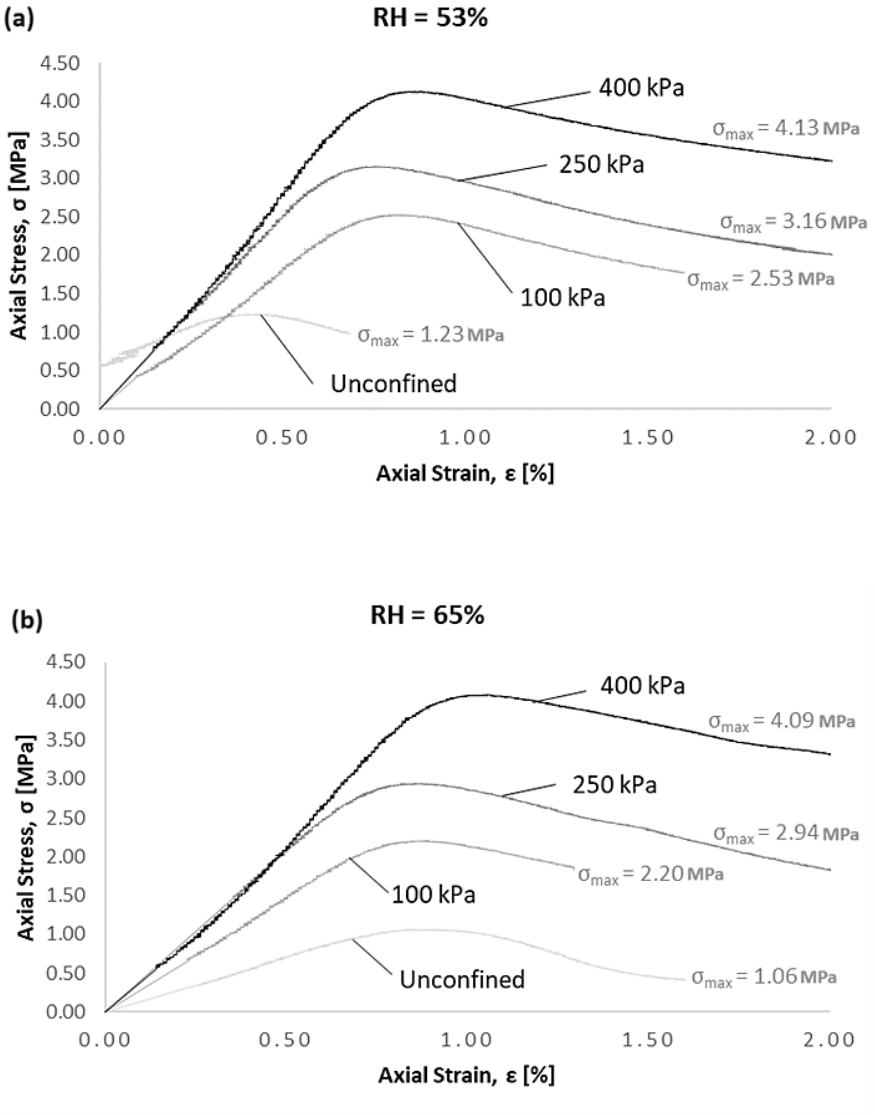


Fig. 5 Stress-strain curves obtained from triaxial tests at varying confining pressures on accelerated carbonated samples after 30 days of curing at 65% RH

Fig. 6 (a-c) shows the stress-strain curves obtained from triaxial tests on samples which were naturally carbonated at the three different relative humidity levels of 53%, 65% and 95% for 100 days. Instead, Fig. 7 shows the stress-strain curves from triaxial

tests on accelerated carbonated samples at a single relative humidity of 65% for 100 days. As before, a slightly lower strengths were measured for the accelerated carbonated samples compared to the naturally carbonated ones.

Comparisons between Fig. 4 and Fig. 6 indicate that, the naturally carbonated samples cured for 100 days exhibit lower peak strengths than those cured for 30 days, though the difference reduces with increasing confining pressure. This is clearly shown in Fig. 8, which summarizes the evolution of peak strength with relative humidity for the two curing times of 30 and 100 days.



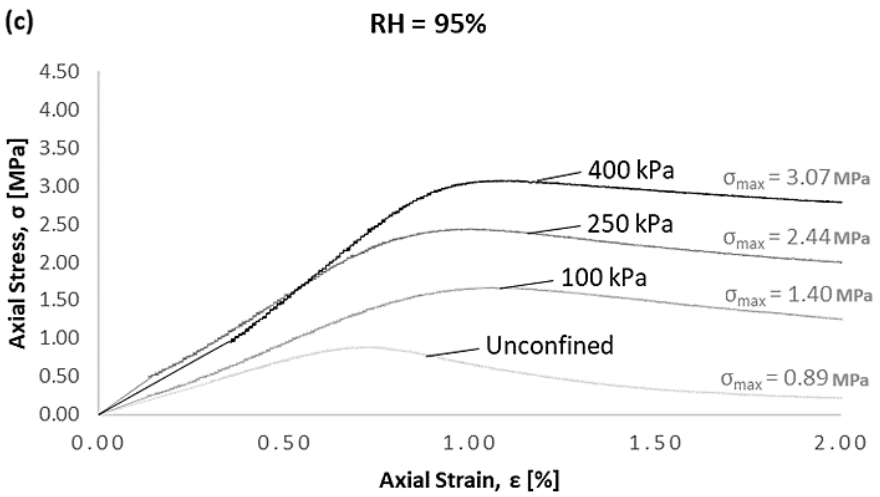


Fig. 6 Stress-strain curves obtained from triaxial tests at varying confining pressures on naturally carbonated samples: after oven-drying (a), after 100 days of curing at 53% (b), 65% (c) and 95% (d) RH

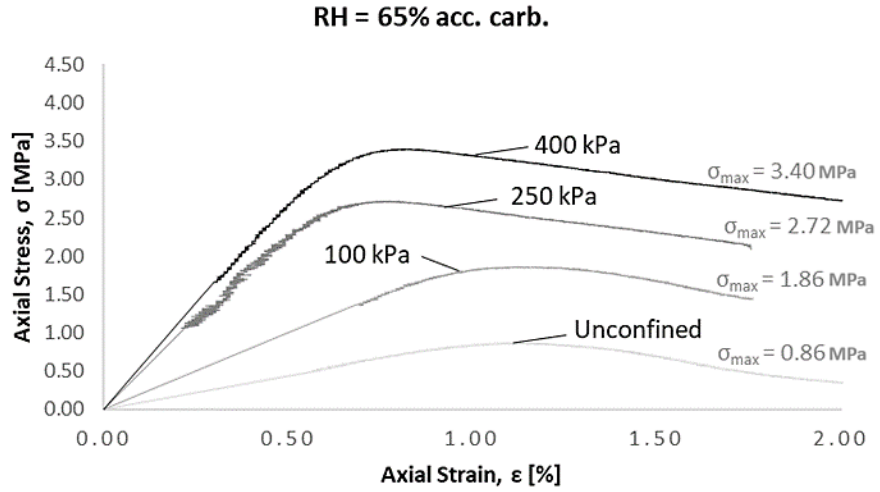


Fig. 7 Stress-strain curves obtained from triaxial tests at varying confining pressures on accelerated carbonated samples after 100 days of curing at 65% RH

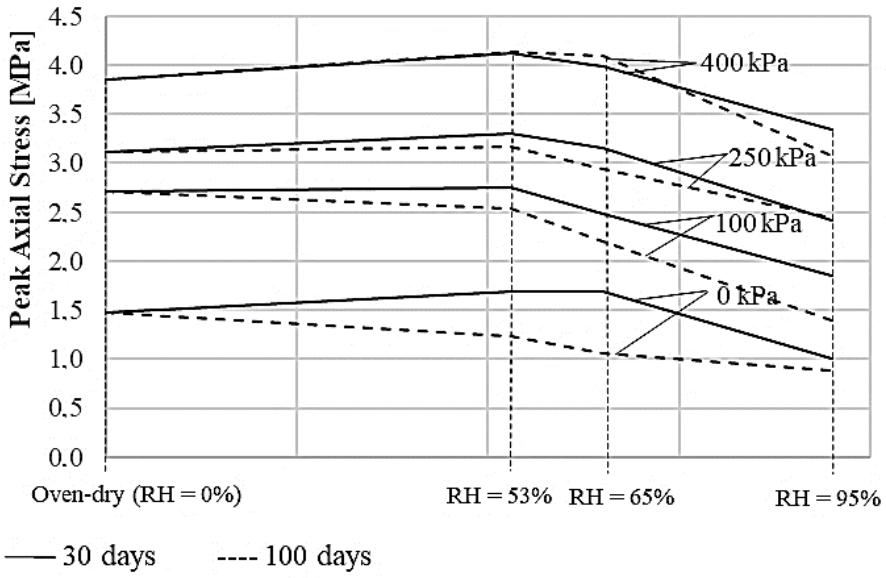


Fig. 8 Evolution of peak strength with relative humidity at varying confining pressures after natural carbonation during 30 or 100 days

The lower strength of the samples cured for 100 days compared to those cured for 30 days is unexpected, given their lower degree of saturation (Table 1), which should result in stronger inter-particle capillary bonds and, consequently, higher strength. Carbonation therefore seems to be the only phenomenon that can explain the progressive decrease in strength supporting the previous observations made for samples subjected to accelerated carbonation. Following the initial strength gain during curing due to the residual hydration reactions and carbonation of the portlandite $Ca(OH)_2$ phase, strength decreases over the long term as the calcium silicate hydrate C-S-H gel undergoes carbonation. Hou *et al.* [25] notably demonstrated that carbonation continues over time, with the recycled material persistently absorbing CO_2 in the long term, leading to further precipitation of $CaCO_3$. The authors attributed this ongoing $CaCO_3$ formation to the decalcification of the calcium silicate hydrate (C-S-H) gel, which ultimately causes a weakening of the material. While the carbonation of portlandite could increase compressive strength, the carbonation of C-S-H has the opposite effect.

Inspection of Fig. 8 also indicates that, for both curing times and for all four confining stresses, the peak strength is highest at RH=53% while it decreases with increasing relative humidity and, hence, increasing degree of saturation. This is mostly due to the smaller number of stabilizing inter-particle capillary bridges at higher saturation levels and, to a lesser degree, to the lower diffusion of carbon dioxide through water filled pores, which limits the carbonation reaction.

3.2. Modelling of strength and stiffness

The results from the previous tests were processed to model the variation of both peak strength and initial Young's modulus with confining pressure at different relative humidity and carbonation levels. The initial Young's modulus E [MPa] was measured as the slope of the axial stress-strain curves over the stress range up to 20% of peak strength where the material response is reasonably linear. However, given the granular and partially cemented nature of the recycled aggregates, some irreversible (plastic) deformations may occur even at low stress levels. Therefore, the calculated stiffness does not represent a purely elastic response and is more accurately described as an "apparent" Young's modulus, reflecting the initial tangent stiffness of the material under the given loading conditions. The measured values of peak stress were linearly interpolated, for each set of samples, in the plane of deviatoric stress q versus mean stress p by the Mohr-Coulomb failure equation for cohesive-frictional soils:

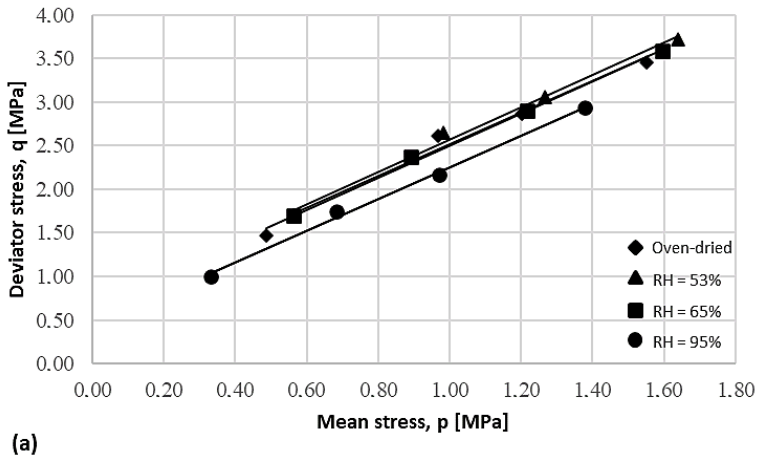
$$\mathrm{q} = \mathrm{C} + \mathrm{M}\,\mathrm{p} \tag{2}$$

where the coefficients C [MPa] and M [-] are respectively the intercept and slope of the failure envelope. These coefficients were then converted into the corresponding values of cohesion c' [MPa] and friction angle φ' [°] by means of the following equations:

$$M = \frac{6\,\sin\varphi'}{3 - \sin\varphi'} \quad \rightarrow \quad \sin\varphi' = \frac{3\,M}{6 + M} \tag{3}$$

$$C = \frac{6\,c\,\cos\varphi'}{3 - \sin\varphi'} \quad \rightarrow \quad c' = \frac{(3 - \sin\varphi')\,C}{6\,\cos\varphi'} \tag{4}$$

Fig. 9a plots the peak deviator stress q, measured under different confinement levels, against the corresponding mean stress p for the naturally carbonated samples after oven-drying or after 30 days of curing at the relative humidity levels of 53%, 65% and 95%. Fig. 9b shows a similar plot for the naturally and accelerated carbonated samples after 30 days of curing at the relative humidity of 65%.



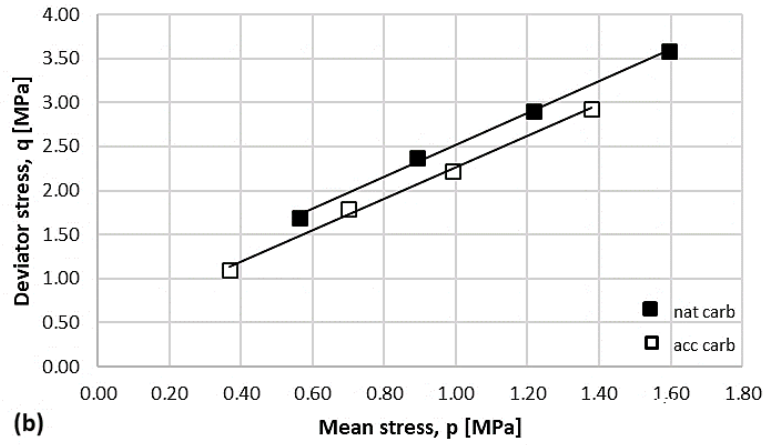
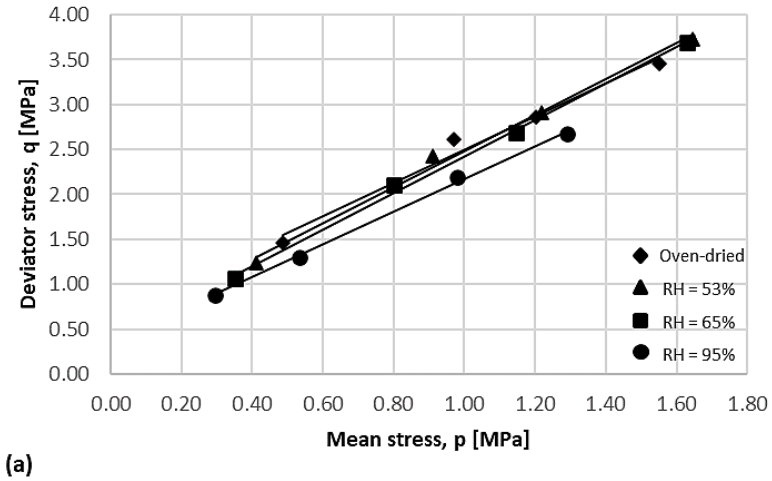


Fig. 9 Peak strength envelopes: naturally carbonated samples after oven-drying or 30 days of curing at 53%, 65% and 95% RH (a); naturally vs. accelerated carbonated samples after 30 days of curing at 65% RH (b)

Fig. 10a plots the peak deviator stress q, measured under different confinement levels, against the corresponding mean stress p for the naturally carbonated samples after oven-drying or after 100 days of curing at relative humidity levels of 53%, 65% and 95%. Fig. 10b shows instead a similar plot for the naturally and accelerated carbonated samples after 100 days of curing at the relative humidity of 65%.

The interpolation of the data in Figs. 9 and 10 is summarized in Table 2, which presents the strength parameters of the material cured for different times at distinct levels of relative humidity and carbonation.



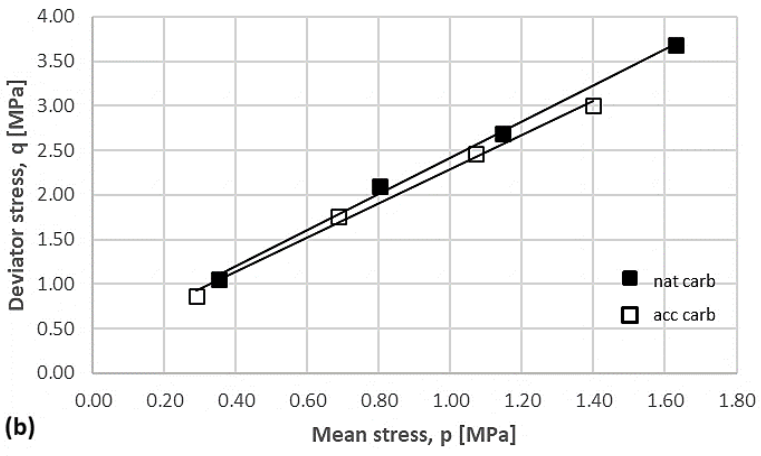


Fig. 10 Peak strength envelopes: naturally carbonated samples after oven-drying or 100 days of curing at 53%, 65% and 95% RH (a); naturally vs. accelerated carbonated samples after 100 days of curing at 65% RH (b)

Table 2 Strength parameters of naturally and accelerated carbonated after oven-drying or 30 and 100 days of curing at 53%, 65% and 95% RH

	M *[-]*	φ *[°]*	C *[-]*	c *[MPA]*
30 days				
RH = 53%	1.86	45.1	0.71	0.39
RH = 65% (nat. carb.)	1.81	44.1	0.70	0.38
RH = 65% (acc. carb.)	1.78	43.4	0.48	0.26
RH = 95%	1.82	44.35	0.43	0.23
100 days				
RH = 53%	2.00	48.7	0.48	0.27
RH = 65% (nat. carb.)	2.03	49.4	0.39	0.22
RH = 65% (acc. carb.)	1.92	46.58	0.37	0.20
RH = 95%	1.83	44.42	0.35	0.19
Oven-dried				
RH = 0%	**1.85**	**45.0**	**0.65**	**0.35**

Fig. 9 and Table 2 show moderately higher levels of friction angle and cohesion for the samples that have been naturally carbonated for 30 days at a relative humidity of 53% compared to oven-dried samples. However, any further increase of relative humidity for the same curing time results in lower strength levels. This behavior may be explained by the capillary bonding between particles, which attains a maximum around a relative humidity of 53% when the number of inter-particle water menisci generating extra strength is highest. Dryer or wetter conditions correspond to lower strength levels because, as the material desiccates or soaks with respect to the relative humidity of 53%, the inter-particle menisci start to vanish. The shear strength parameters also depend on hydraulic reactivity and carbonation. In particular, carbonation reaction is delayed in samples exposed to high relative humidities because the higher water contents prevent the diffusion of CO_2 in the porous media. Delayed carbonation reaction and less inter-particle water menisci might explain the

lower values of cohesion obtained for samples equalized at relative humidity of 95% after only 30 days of curing. Contrary to intuition, accelerated carbonation produces a clear decrease of strength, which is more evident in terms of cohesion than friction angle and could be caused by the detrimental carbonation of the calcium silicate hydrate (C-S-H) gel as previously discussed.

Inspection of Fig. 10 and Table 2 shows similar trends for the samples that have been cured for 100 days. In general, the friction angle is higher and the cohesion is lower if compared to the samples that have been carbonated for 30 days. Interestingly, the values of friction angle and cohesion are very similar after 30 and 100 days of curing at the relative humidity of 95%. Therefore, the strength characteristics of the material appear differently influenced by pore moisture at distinct saturation levels. An explanation for this behavior could be provided by the varying effect of bulk and meniscus water on the interaction among material particles, including direct contact, meniscus contact, and cement contact. Furthermore, once again, the carbonation reaction may play a role. For samples equalized at relative humidity of 95% the carbonation reaction is delayed while for samples equalized at lower relative humidities the carbonation reaction involves the C-S-H resulting in lower cohesion.

Fig. 11a shows the variation of the apparent Young's modulus E with confining pressure σ_c of naturally carbonated samples after oven-drying or after 30 days of curing at relative humidity levels of 53%, 65% and 95%. Inspection of Fig. 11a indicates that the initial apparent Young's modulus ranges between 150 MPa and 600 MPa and it decreases with both increasing relative humidity, as a consequence of higher water saturation, and reducing confinement. Fig. 11b shows instead the variation of the

apparent Young's modulus E with confining pressure σ_c for the samples that have been naturally or accelerated carbonated during 30 days at the relative humidity of 65%.

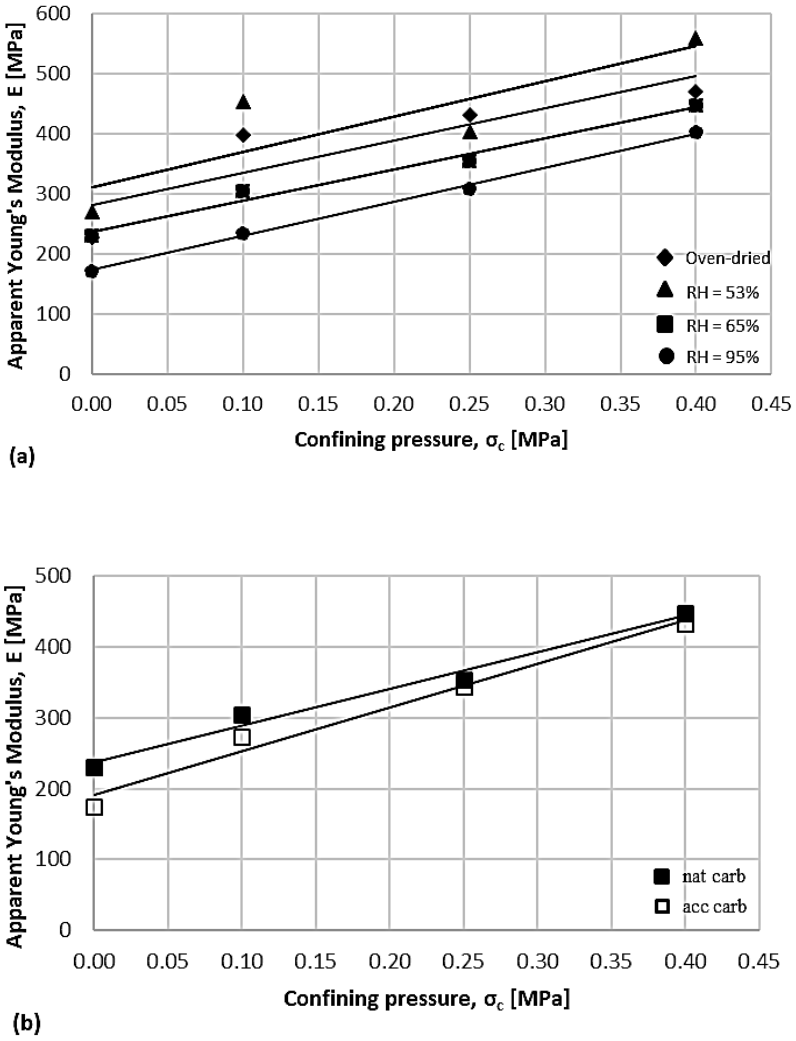


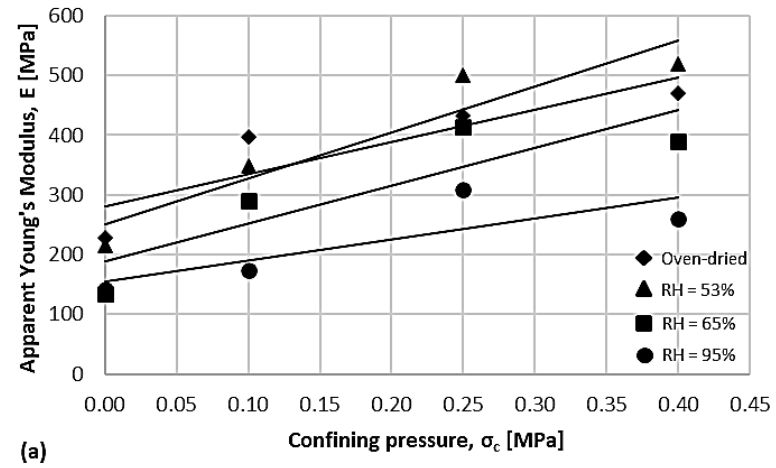
Fig. 11 Apparent Young's Modulus vs. confining pressure: naturally carbonated samples after oven-drying or 30 days of curing at 53%, 65% and 95% RH (a); naturally vs. accelerated carbonated samples after 30 days of curing at 65% RH (b)

Fig. 12a shows the variation of the apparent Young's modulus E with confining pressure σ_c for naturally carbonated samples after oven-drying or after 100 days of curing at relative humidity levels of 53%, 65% and 95%. Fig. 12b shows instead the variation of the apparent Young's modulus E with confining pressure σ_c for the samples treated by natural or accelerated carbonation for 100 days at the relative humidity of 65%.

Interestingly, the comparison between Fig. 11a and Fig. 12a indicates that stiffness exhibits a similar dependency on relative humidity and confining pressure after both 30 and the 100 days of curing. The sensitivity of material stiffness to curing time appears therefore lower than that of peak strength, which showed a clear decline when curing time increased from 30 to 100 days. The reasons of this difference may be found in the different influence that the carbonation of the calcium silicate hydrate C-S-H gel has on stiffness and peak strength, respectively.

Inspection of Fig. 11b and Fig. 12b similarly suggests that accelerated carbonation does not noticeably changes the stiffness of the material. Only for largest confinement pressure of 400 kPa, there is a noticeable difference of stiffness between naturally and accelerated carbonated samples after 100 days of curing. Yet, even in this case, the difference might reflect potential inaccuracies associated to the determination of the apparent Young's modulus rather than real material behavior.

Overall, the above results indicate that further studies must be carried out to better appreciate the impact of carbonation on the evolution of the material microstructure. This includes a deeper understanding of the unstable nature of the calcium carbonate polymorphs (e.g. aragonite and vaterite) which are precipitated as a consequence of the carbonation of the C-S-H gel. Another aspect deserving further investigation is the interplay between the effects of pores clogging, carbonation shrinkage and induced cracking.



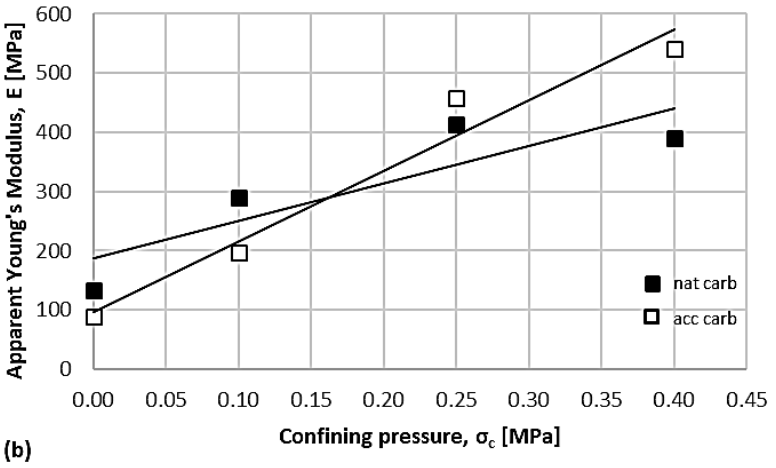


Fig. 12 Apparent Young's Modulus vs. confining pressure: naturally carbonated samples after oven-drying or 100 days of curing at 53%, 65% and 95% RH (a); naturally and accelerated carbonated samples after 100 days of curing at 65% RH (b)

4 Conclusions and recommendations

Recycled aggregates (RA) exhibit measurable hydraulic activity, making them particularly attractive for geotechnical applications such as road pavement base layers. Their reuse significantly reduces the exploitation of natural aggregates and diverts substantial volumes of construction and demolition waste from landfills, promoting a circular economy and supporting sustainability goals in civil infrastructure. This study evaluated the mechanical behavior of compacted fine recycled aggregates cured

under different environmental conditions, particularly varying levels of relative humidity and carbon dioxide concentration. Results showed that:

- peak cohesion increases from approximately 20 kPa to 35 kPa and friction angle from 36° to 42° as relative humidity decreases from 95% to 53%, confirming the influence of capillary suction at lower water contents. The reduction in water saturation enhances inter-particle bonding and facilitates CO_2 diffusion through the pore network, promoting carbonation reactions. However, ductility also increased at higher humidity levels, highlighting a trade-off between strength and deformation capacity depending on moisture availability. These findings suggest that optimizing moisture conditions at the time of compaction is critical: lower saturation favors strength development, while slightly higher saturation may be desirable where enhanced deformability is required;
- regarding carbonation, the study found that accelerated carbonation at 3% CO_2 consistently resulted in lower peak strength values - by approximately 15% to 25% - compared to natural carbonation at 0.05% CO_2, particularly after 100 days of curing. This strength reduction is attributed to the complete consumption of portlandite ($Ca(OH)_2$), which shifts carbonation to the calcium silicate hydrate (C-S-H) phases, known to weaken the cementitious matrix. These findings suggest that natural carbonation over 30 days provides better strength development than extended or accelerated carbonation regimes, emphasizing the need for careful curing protocol selection in field applications. Further investigation into the evolution of microstructure and the nature of the calcium carbonate polymorphs formed during carbonation would be beneficial for optimizing long-term performance;

- Fracture patterns observed in triaxial tests reveal that failure often initiates at the aggregate - mortar interface, indicating that this transition zone is a structural weak point in RA materials. Future design approaches should account for this interface as a critical structural feature, and materials engineering strategies aiming to improve interface cohesion - such as selective grading or surface modification - could enhance the overall performance of recycled aggregates;
- The stiffness of the material, measured as initial apparent Young's modulus, ranged between 150 MPa and 600 MPa, increasing with confining pressure and decreasing relative humidity. This stiffness was found to stabilize after 30 days of curing and was largely unaffected by CO_2 concentration, suggesting that early-age measurements can reliably inform long-term pavement design.

In practical terms, these results highlight that RA-based pavement layers perform best when compacted at controlled, relatively low moisture contents. Moisture control during placement is essential to promote strength development via suction and ensure long-term stability. Additionally, exposure to ambient atmospheric carbonation over moderate timeframes (e.g., 30 days) is preferable to forced carbonation strategies, particularly in unbound or lightly bound base layers. In real-world pavement conditions, where exposure to cycles of drying, wetting, and limited carbonation is common, these findings support the use of RA as a technically and environmentally viable material, provided that moisture conditions during construction and service life are carefully managed.

Ultimately, the study reinforces the need to integrate environmental curing conditions into pavement design frameworks when using recycled aggregates, and it provides

foundational data for setting performance-based specifications in sustainable road construction.

Data availability statement

All data are available from the corresponding author upon reasonable request.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

References

[1] Rao, A., Jha, K. N., & Misra, S. (2007). Use of aggregates from recycled construction and demolition waste in concrete. *Resources, conservation and Recycling*, 50(1), 71-81.

[2] Akhtar, A., & Sarmah, A. K. (2018). Construction and demolition waste generation and properties of recycled aggregate concrete: A global perspective. *Journal of Cleaner Production,* 186, 262-281.

[3] Chauvet-Peyrard, A., & Montérémal, M. (2018). Le recyclage des déchets produits par l'activité de BTP en 2014.

[4] Lancieri, F., Marradi, A., & Mannucci, S. (2006). C&D waste for road construction: long time performance of roads constructed using recycled aggregate for unbound pavement layers. WIT Transactions on Ecology and the Environment, 92.

[5] Jayakody Arachchige, S. P., Gallage, C., & Kumar, A. (2012). Assessment of recycled concrete aggregates for road base and sub-base. In Proceedings of the second international conference on geotechnique, construction materials and environment (pp. 575-579). The GEOMATE International Society.

[6] Al-Mosawe, H., Albayati, A., Wang, Y., & Mashaan, N. S. (2022). An Experimental Study of Granular Material Using Recycled Concrete Waste for Pavement Roadbed Construction. Buildings 2022, 12, 1926.

[7] Cadenas Alvarado, L. Y., Jacinto Huamanchumo, R. M., Garcia Chumacero, J. M., Salinas Vásquez, N. R., & Chavez Cotrina, C. O. (2024). Experimental study of hybrid concrete blocks based on sawdust and calcined clay using cement as a binder. Innovative Infrastructure Solutions, 9(11), 445.

[8] Muñoz Pérez, S. P., Pardo Becerra, J. M., García Chumacero, J. M., Sánchez Diaz, E., Diaz Ortiz, E. A., Laffite, E. D. R., ... & Briceño Mendoza, Y. M. (2024). Evaluation of the physical and mechanical properties of concrete with the incorporation of recycled concrete aggregate. Innovative Infrastructure Solutions, 9(6), 204.

[9] García Chumacero, J. M., Gonzales Macedo, J. L., & Sánchez Castillo, D. J. (2024). Contribution of agricultural ashes and HDPE as a waste material for a sustainable environment applied to the stabilization of a low plasticity clay soil. Innovative Infrastructure Solutions, 9(3), 67.

[10] Akbarnezhad, A., Ong, K. C. G., Tam, C. T., & Zhang, M. H. (2013). Effects of the parent concrete properties and crushing procedure on the properties of coarse recycled concrete aggregates. *Journal of Materials in Civil Engineering*, *25*(12), 1795-1802.

[11] Cazacliu, B., Sampaio, C. H., Miltzarek, G., Petter, C., Le Guen, L., Paranhos, R., ... & Kirchheim, A. P. (2014). The potential of using air jigging to sort recycled aggregates. *Journal of Cleaner Production*, *66*, 46-53.

[12] Paranhos, R. S., Cazacliu, B. G., Sampaio, C. H., Petter, C. O., Neto, R. O., & Huchet, F. (2016). A sorting method to value recycled concrete. *Journal of Cleaner Production*, *112*, 2249-2258.
[13] Amario, M., Rangel, C. S., Pepe, M., & Toledo Filho, R. D. (2017). Optimization of normal and high strength recycled aggregate concrete mixtures by using packing model. *Cement and Concrete Composites*, *84*, 83-92.
[14] Ambros, W. M., Sampaio, C. H., Cazacliu, B. G., Miltzarek, G. L., & Miranda, L. R. (2017). Usage of air jigging for multi-component separation of construction and demolition waste. *Waste management*, *60*, 75-83.
[15] Hoong, J. D. L. H., Lux, J., Mahieux, P. Y., et al. (2020). Determination of the composition of recycled aggregates using a deep learning-based image analysis. *Automation in Construction*, 116, 103204.
[16] Hou, Y. (2021b). Contribution au développement de la valorisation des déchets inertes du BTP: Étude de la carbonatation des granulats recyclés compactés (Doctoral dissertation, Université de La Rochelle).
[17] Cheng, H., Wang, Y., Liu, J., Poon, C. S., Ren, P., Liu, Y., & Chen, Z. (2024). The long term re-cementation of recycled concrete aggregate in road sub-base and its impacts on pavement performance. Transportation Geotechnics, 49, 101431.
[18] Poon, C. S., Qiao, X. C., & Chan, D. (2006). The cause and influence of self-cementing properties of fine recycled concrete aggregates on the properties of unbound sub-base. Waste management, 26(10), 1166-1172.
[19] Vegas, I., Ibañez, J. A., Lisbona, A., De Cortazar, A. S., & Frías, M. (2011). Pre-normative research on the use of mixed recycled aggregates in unbound road sections. *Construction and Building Materials*, 25(5), 2674-2682.
[20] Chai, L., Monismith, C. L., & Harvey, J. O. H. N. (2009). Re-Cementation of Crushed Material in Pavement Bases.
[21] Paige-Green, P. (2010). Preliminary evaluation of the reuse of cementitious materials.
[22] Jitsangiam, P., Suwan, T., Pimraksa, K., Sukontasukkul, P., & Chindaprasirt, P. (2021). Challenge of adopting relatively low strength and self-cured geopolymer for road construction application: a review and primary laboratory study. International Journal of Pavement Engineering, 22(11), 1454-1468.
[23] Arm, M. (2001). Self-cementing properties of crushed demolished concrete in unbound layers: results from triaxial tests and field tests. *Waste Management*, 21(3), 235-239.**[24]** Hou, Y., Ji, X., & Su, X. (2017). Mechanical properties and strength criteria of cement-stabilised recycled concrete aggregate. *International Journal of Pavement Engineering*, *20*(3), 339-348.
[24] Hou, Y., Ji, X., & Su, X. (2017). Mechanical properties and strength criteria of cement-stabilised recycled concrete aggregate. *International Journal of Pavement Engineering*, *20*(3), 339-348.
[25] Hou, Y., Mahieux, P. Y., Lux, J., Turcry, P. & Abdelkarim, A. I. T. (2019) Qualification of the residual reactivity of compacted recycled aggregates.
[26] Hou, Y., Mahieux, P.-Y., Lux, J., Turcry, P., Aït-Mokhtar, A. (2021a). Carbonation rate of compacted recycled aggregates for sub-base layers of pavement. Constr. Build. Mater. 312, 125420.
[27] NF EN 933-11. (2009). Tests for geometrical properties of aggregates – Part 11: classification test for the constituents of coarse recycled aggregate.
[28] Cerema, Guide d'application, Acceptabilité environnementale de matériaux alternatifs en technique routière, les matériaux de déconstruction issus du BTP, 2016.
[29] NF EN 13286-53. Unbound and hydraulically bound mixtures – Pat 53: methods for the manufacture of test specimens of hydraulically bound mixtures using axial compression.
[30] NF P94-102-1. (2001) Sols: Reconnaissance et essais - Sol traite´ au liant hydraulique, éventuellement associe´ a` la chaux, pour utilisation en couche de forme - Partie 1: définition - Composition – Classification.
[31] LCPC et SETRA, Guide d'application, Traitement des sols à la chaux et/ou aux liants hydrauliques : application à la réalisation des remblais et des couches de forme, 2000.

[32] NF P94-050. (1995). Determination of moisture content. Oven drying method, 1995, AFNOR: Paris France.
[33] Tarantino A. (2007). A possible critical state framework for unsaturated compacted soils. Géotechnique, 57(4), 385–389.
[34] Wheeler, S. J., & Karube, D. (1996). Constitutive modelling.
[35] Xiao, J., Li, L., Shen, L., et al. (2015). Effects of strain rate on mechanical behavior of modeled recycled aggregate concrete under uniaxial compression. Construction and Building Materials, (93), 214-222.
[36] Gallipoli D, Gens A, Sharma R, and Vaunat J. (2003). An elasto-plastic model for unsaturated soil incorporating the effects of suction and degree of saturation on mechanical behaviour. Géotechnique, 53(1), 123–136.
[37] Beckett, C. T. S., & Augarde, C. E. (2012). The effect of humidity and temperature on the compressive strength of rammed earth. In Proceedings of 2nd European Conference on Unsaturated Soils (pp. 287-292). Naples, Italy, 20-22 June 2012, Springer, ISBN: 978-3-642-31342-4.
[38] Fredlund, D. G., & Rahardjo, H. (1993). Soil mechanics for unsaturated soils. John Wiley & Sons.
[39] Beal, N. S. (2013). What is the total suction of soil at Oven Dry. Autralian Geomechanics, 48(1), 157-168.
[40] Cuccurullo, A., Gallipoli, D., Bruno, A. W., La Borderie, C., Augarde, C., & Hughes, P. (2018). The effect of ambient humidity on the stiffness and strength of a hyper-compacted silty clay for earth building. In *Proc 7th Int Conf on Unsaturated Soils*.
[41] Etxeberria, M., Vázquez, E., Marí, A., & Barra, M. (2007). Influence of amount of recycled coarse aggregates and production process on properties of recycled aggregate concrete. Cement and concrete research, 37(5), 735-742.
[42] Poon, C. S., Shui, Z. H., & Lam, L. (2004). Effect of microstructure of ITZ on compressive strength of concrete prepared with recycled aggregates. Construction and building materials, 18(6), 461-468.
[43] Grondin, F., Guo, M., Rozière, E., & Loukili, A. (2019). Failure risk of recycled aggregates concrete. In MATEC Web of Conferences (Vol. 281, p. 01017). EDP Sciences.
[44] Shi, C., Liu, M., He, P., & Ou, Z. (2012). Factors affecting kinetics of CO2 curing of concrete. *Journal of Sustainable Cement-Based Materials*, *1*(1-2), 24-33.
[45] Zhang, J., Shi, C., Li, Y., Pan, X., Poon, C. S., & Xie, Z. (2015). Performance enhancement of recycled concrete aggregates through carbonation. Journal of Materials in Civil Engineering, 27(11), 04015029.
[46] Kobayashi, K. (1991). Carbonation of concrete. J. Mater. Concr. Struct. Pavements, 433, pp. 1-14.
[47] Cahyadi, J. H. & Uomoto, T. (1995). Relation between phase composition, pore structure and compressive strength of carbonated mortar. J. Mater. Concr. Struct. Pavements, 526, pp. 133-141.
[48] Nakarai, K. & Yoshida, T. (2015). Effect of carbonation on strength development of cement-treated Toyoura silica sand. Soils and Foundations, (55)4, 857-865.
[49] Pade, C., & Guimaraes, M. (2007). The CO2 uptake of concrete in a 100 year perspective. *Cement and concrete research*, *37*(9), 1348-1356.