CO_2 CAPTURE AND STORAGE IN CONCRETE GENERATED THROUGH EARLY-AGE CABONATION OF CALCIUM SILICATE HYDRATE

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ABSTRACT: Recent global research on the sequestration and definitive storage of CO_2 into concrete due to untimely carbonation of hydrated Ca_2SiO_4 (known as C-S-H), the most important phase of ordinary cement, has piqued the interest of authors of this paper. Limiting the gas footprint released into the atmosphere remains a very topical objective in the world and implicitly, also of the authors. Sodium bicarbonate ($NaHCO_3$) added into starting mix as a partial substitute for cement has already been identified as the suitable option to reach the objective. Another very interesting effect has been experimentally highlighted in the direction of increasing the mechanical resistance particularly, at an early-age of the concrete preparation. Specific measurements carried out after only 6 hours has found very high values of compression and flexural strength, close to the values corresponding to the end of concrete curing process of 28 days. This procedure has not yet been finalized in technological terms in the world. Its importance lies in the possibility of manufacturing without CO_2 emissions and utilizing the traditional Portland cement as the main binder for construction concrete in the current situation of world environmental crisis.
KEY WORDS: CO_2 sequestration, C-S-H, untimely carbonation, sodium bicarbonate, cement, mechanical strength.

1. INTRODUCTION

The tendency for the Earth's atmosphere to overheat due to excessive greenhouse gas emissions has greatly affected the industrial manufacture of cement, which has been the traditional binder for over 100 years of the construction concrete. Under these conditions, the need arose to guide researchers and manufacturers in this field towards other types of cementitious materials with pozzolanic abilities that could replace ordinary Portland cement. Especially, industrial alumina-silicate by-products (carbon ash, industrial slag, rice ash, mud from bauxite processing, etc.) have experimentally proven appropriate in this direction [1]. French inventor J. Davidovits proposed and claimed the use of alumina-silicate materials for the manufacture of so-called geopolymers, including geopolymer concretes [2, 3].

Recently, the interest of researchers has been focused on CO_2 capture and storage in concrete following the untimely carbonation of Ca_2SiO_4 hydrated. C-S-H is the principal binding element of the Portland binder, according to [4]. Through hydration, the cement phases called alite (C_3S) and belite (C_2S), respectively, constituting approximately 3/4 of the cement mass, have a major impact on cement features (strength and setting time) [5, 6].

The literature registers several investigations of the CO_2 storage capacity of concrete [7-9]. In the paper [7], the kinetics of C-S-H carbonation as the main component of cementitious materials was analyzed and the properties evolution of the carbonation products was also investigated. C-S-H carbonation occurs in three stages: dissolution, diffusion, and slow reaction. In the last stage, C-S-H fully decomposes and the mix of calcium carbonate in microcrystalline state and modified calcium in silica gel becomes the final material of the process. The work provided new knowledge on sequestration of carbon dioxide using C-S-H phase included in cementitious substances.

According to [8], carbonation acceleration of recycled concrete aggregates was performed at 0.1 and 5 bars, respectively. The outcomes indicated that the use of aggregates in the carbonation state into concrete products favoured strength growing of 2-18 %. Also, the use of carbonated recycled aggregate led to reduction in drying shrinkage. Carbonation performed at high pressure (5 bar) and long duration (24 hours) did not indicate any influence on the

concrete properties. It was shown that the best efficiency of carbonated concrete manufacturing is the use of low pressure (0.1 bar) and a short duration (6 hours).

In accordance with the conclusions of the work [9], although several projects for the development of carbon capture technologies are known, applying the CO_2 mineralization in manufacturing the Portland cement is less advanced. However, the connection between direct CO_2 mineralization techniques with the possibility of concrete recycling shows the capability of significantly reducing CO_2 emissions in concrete manufacturing. Analysis of reactions that occur during CO_2 mineralization showed that the procedures can occur at usual levels of temperature and pressure with the possibility of advanced CO_2 sequestration.

The work presented below aimed to experimentally making a high-strength concrete under the conditions of using the pre-hardening technique of the fresh mixture. $NaHCO_3$ was chosen as a limited Portland cement replacing (in the range of 10-23 %). The work wishes to contribute for improving knowledge in the field of the new procedure of untimely concrete hardening in association with carbon dioxide sequestration.

2. PROCEDURES AND MATERIALS

2.1 Procedures

It is known that the carbonation process has the effect of reducing porousness due to the difference between the molar dimension of hydration materials and the formed calcium carbonate. A porousness reduction was observed after the completion of carbonation of „CEM I'' cement-based products both in situ and under accelerated exposure conditions. Carbonation of C-S-H phase could be a motivation of the porousness decrease. Researchers that investigated this carbonation process have accepted the idea of a complex process existence of C-S-H decalcification and polymerization as well as the generation of silica gel as an amorphous product [10], in accordance with the reaction (1).

$$C_xS_yH_z + xC \rightarrow xC\bar{C} + ySH_t + (z - yt)H \quad (1)$$

where:

$C_xS_yH_z$ – C-S-H; C - CO_2; C$\bar{C}$ - calcium carbonate; SH_t – silica gel; H – H_2O.

The stoichiometric coefficients (x, y, z) of reaction (1) are not fully clarified to date. Also, the water quantity contained in SiO_2 gel is not known and the formed type of $CaCO_3$ (calcite, vaterite, or aragonite). Therefore, the cubic dimension of solid phases is not known, explaining the disconfort in evaluating the porousness generated by C-S-H carbonation.

Porousness as a dependence on the level of silica gel hydration may explain its increasing trend, which was already noted in cementitious processes based on carbon ash or nano-silica, in which C-S-H phase exists [11].

The pre-curing method was chosen for creating the conditions of adequate humidity in the concrete mass, that by turning can facilitate the water absorption into the formed cells of carbon dioxide. Mineral carbonation (also called CO_2 mineralization) is a procedure of capturing the carbon dioxide bond for creating carbonates. As it is known, these are very poorly soluble into H_2O and practically, in thermodynamic terms, stable under normal atmospheric conditions [12]. Thus, the conditions for the definitive storage of CO_2 are created. Early-age CO_2 binding procedure has the ability to sequester CO_2 through its mineralization and at the same time, to use the reaction products ($CaCO_3$, SiO_2 gel, or decalcified C-S-H phase) ensuring the improvement of untimely resistance of the final product [9].

In ordinary Portland cementitious material, calcium silicates slowly hydrate, poorly hydrate Ca_2SiO_4 making C-S-H as nanoparticles, which spread between the pores, crystallizing. $NaHCO_3$ utilizing method allows that calcium carbonate ions available in the hydration zones facilitate turning nanoparticles into amorphous materials, comporting as a nucleation zone for forming the C-S-H phase. Further transformations can perform a composite having in its composition $CaCO_3$ and C-S-H and favour growing the untimely mechanical resistance. The advantageous results of carbonation during the fresh mixing indicate a permanent storage location for carbon dioxide in the composite volume.

2.2 Materials

The experiment has constituted a comparison between a reference Portland cement-based concrete sample and four experimental versions of concrete specimens manufactured by partially replacing the cement with sodium bicarbonate ($NaHCO_3$).

Materials used for preparing the reference sample were: „CEM I" type Portland cement (350 kg·m^{-3}), carbon ash (4.5 kg·m^{-3}), processed slag (5.2 kg·m^{-3}), and silica fume under 150 nm (4.3 kg·m^{-3}), as additional cementitious materials. Coal ash and granulated blast furnace slag, representing by-products of the energy industry and metallurgy industry, respectively, were supplied approximately 10 years ago by Paroseni-Thermal power plant and

ArcelorMittal Galati (Romania). In order to use them in this experiment, the two industrial by-products were ball milled and sieved, the selected particle size being below 90 μm. Silica fume as a by-product of silicon metallurgy is commercially available at very low particle size, being used in low amounts for concrete manufacturing due to its ability to increase strength and workability. Mainly this product is supplied by companies from the United States and Canada. The aggregate was composed of fine river sand with particle size under 2.3 mm (590 $kg \cdot m^{-3}$) and crushed granite stone with grain size under 18 mm (853 $kg \cdot m^{-3}$). The H_2O amount was 182 $kg \cdot m^{-3}$, under conditions of water/cement report of 0.52 to be within the limits suitable for good resistance and robustness of concrete. The fresh concrete was curing through its usual pouring into a mould and keeping at 75 °C for 24 hours in an electric oven. After removing from the mould, the concrete specimen was stored at ambient thermal conditions for 28 days prior to determining its corresponding characteristics.

The raw material compositions chosen for this test are shown in Table 1.

Experimental versions adopted for making early-age carbonation and CO_2 capture into concrete were based on the $NaHCO_3$ utilization as a partial cement substitute in preparing mixture within the limits of 10-23 %. $NaHCO_3$ repartition in the four experimental mixtures is shown in Table 2.

Table 1. Composition of materials

Composition (wt. %)	CEM I cement	Ash	Slag	Silica fume	River sand	Granite stone
SiO_2	20.2	54.4	37.4	92.6	97.2	73.8
Al_2O_3	5.5	26.5	6.4	0.9	1.1	12.7
Fe_2O_3 (FeO)	4.1	4.8	6.9	2.0	0.3	(2.1)
CaO	65.4	3.5	39.9	1.0	0.03	1.1
MgO	0.7	2.5	3.5	0.9		0.7
Na_2O	0.3	0.4	0.1	-	0.13	3.8
K_2O	-	0.6	0.3	1.4		3.8
MnO	-	-	2.1	0.1	-	-
TiO_2	-	-	-	0.2	0.03	-
SO_3	2.6	1.7	-	0.3	-	-
LOI	1.4	-	-	5.0	-	-

Table 2. Composition of variants

Composition ($kg \cdot m^{-3}$)	Variant A	Variant B	Variant C	Variant D
CEM I cement type	315	300	284	269.5
$NaHCO_3$	35 (10.0 %)	50 (14.3 %)	66 (18.9 %)	80.5 (23.0 %)
Carbon ash	4.5	4.5	4.5	4.5
Processed slag	5.2	5.2	5.2	5.2
Silica fume	4.3	4.3	4.3	4.3
River sand	590	590	590	590
Crushed granite stone	853	853	853	853
Working water	182	182	182	182

In accordance with the data in Table 2, the only values that were modified in the four experimental versions were those corresponding to Portland cement and, respectively, $NaHCO_3$. Compared to the reference sample, the value of the cement amount recorded successive decreases of 35, 50, 66, and 80.5 $kg \cdot m^{-3}$, i.e. 10.0; 14.3; 18.9; and 23.0 %, respectively.

2.3 Investigation methods for determining the product features

Apparent denseness was measured using Archimedes' principle in conformity with ASTM C642-21 standard [13]. The porousness was measured using a vacuum saturation procedure according to [14, 15]. The compression resistance of specimens was determined with a hydraulically operated compression testing machine with a pressing capacity of 1000 tons-force, in accordance

with ASTM C133-97 (2015) standard and the flexural strength was determined according to ASTM C78 standard by the use of a beam with third-point-loading. The water uptake of concrete samples was measured by their immersion under water (ASTM C373-18 standard). Microstructural peculiarities of specimens were investigated with ASONA type smartphone microscope.

3. OUTCOMES AND COMMENTS

3.1 Outcomes

Due to the new concrete manufacturing procedure characterized by CO_2 sequestering into its mass, the curing process was much faster, taking place at an early-age when the concrete was still undried, i.e. before pouring the slurry into the mould. The curing process took place at ambient temperature and normal pressure, without additional heat input.

The properties of the specimens produced in the current experiment were compared with the reference concrete sample made by a similar manufacturing recipe (except $NaHCO_3$ which was not used). The physio-heat properties (denseness, porousness, water uptake, and heat conductance) of concrete specimens determined in the current testing process, together with the outcomes of the reference sample, are shown in Table 3. The mechanical features (compression and flexural resistance) of concrete specimens experimentally made were successively measured after 6 hours, 24 hours, 7 days, and 28 days. The reference specimen features were identified at the end of the 7 and 28 curing days, respectively.

Table 3. Physio-heat features of concrete specimens

Feature	Version				Reference specimen (RS)
	A	B	C	D	
Apparent denseness ($kg \cdot m^{-3}$)	2198	2212	2221	2231	2178
Porousness (%)	21.5	21.1	20.7	20.4	20.8
Water uptake (vol. %)	4.4	4.6	4.7	4.9	4.7
Heat conductance ($W \cdot m^{-1} \cdot K^{-1}$)	0.415	0.421	0.426	0.434	0.432

Table 4. Compression and flexure resistance of specimens

Version	Compression resistance (MPa)				Flexure resistance (MPa)			
	6 hours	24 hours	7 days	28 days	6 hours	24 hours	7 days	28 days
A	41.9	42.5	42.8	43.0	8.5	8.7	8.8	8.9
B	43.8	44.2	44.6	44.9	10.1	10.3	10.5	10.6
C	45.5	46.2	46.6	46.7	11.5	11.6	11.8	11.8
D	47.1	47.9	48.9	48.9	12.8	13.0	13.1	13.1
RS	-	-	30.9	43.3	-	-	8.7	11.8

Experimental outcomes included in Table 3 and 4 are interesting in the fresh hardening period terms of concrete specimens. The required curing time has been reduced from late-age of up to 28 days to early-age. Thus, the effects of rapid hardening were felt still during the mixing of component materials. In physio-thermal terms, final characteristics of concrete specimens showed the increasing trend of the apparent denseness (from 2198 to 2231 $kg \cdot m^{-3}$) compared to the reference specimen (2178 $kg \cdot m^{-3}$) and the tendency of porousness decreasing (between 21.5 and 20.4 %) compared to 20.8 %. The heat conductivity of the concrete samples exhibited an increase from 0.415 to 0.434 $W \cdot m^{-1} \cdot K^{-1}$, reaching a value almost similar to that of the reference sample of 0.432 $W \cdot m^{-1} \cdot K^{-1}$. With the reduction of concrete porousness, the water uptake had a slight increasing trend in the range of 4.4-4.9 vol. %, remaining however within the value range of the reference sample (4.7 vol. %).

The obvious change in mechanical properties (compression and flexure resistance) was highlighted through the acceleration of carbonation procedure of C-S-H due to the addition of $NaHCO_3$ (Table 4). The compression resistance reached values very close to its maximum level in mechanical tests performed after only 6 hours. Experimental variants with lower additions of

$NaHCO_3$ (between 10-14.3 %) kept at ambient thermal conditions for most of 28 days led to low growing the compression resistance from 42.8 to 43.8 MPa (in variant A) and from 44.6 to 44.9 MPa (in variant B). Instead, variants with higher additions of $NaHCO_3$ (18.9-23 %) had very small growing, almost negligible, from 46.6 to 46.7 MPa (variant C) and from 48.9 to 48.9 MPa (variant D). After 7 days of traditional hardening, the compression resistance values of the reference specimen reached only 30.9 MPa, by comparison with to the 42.8-48.9 MPa range of the actual test and after 28 days of hardening it reached 43.3 MPa compared to the 43.0-48.9 MPa range recorded by the experimental samples.

The flexure resistance performances had a relatively similar evolution to that of the compression resistance. Very high values (8.5-12.8 MPa) compared to those corresponding to 28 curing days were achieved in the measurements performed for the four variants after 6 hours. The determination of these resistance type values highlighted their almost constant maintenance after longer durations of the curing process. The specimen considered as a reference for comparison was able to reach 8.7 MPa at the end of 7 days and 11.8 MPa at the end of the 28 curing days.

Surface appearance of concrete specimens investigated at early-age (6 hours) is presented in Figure 1.

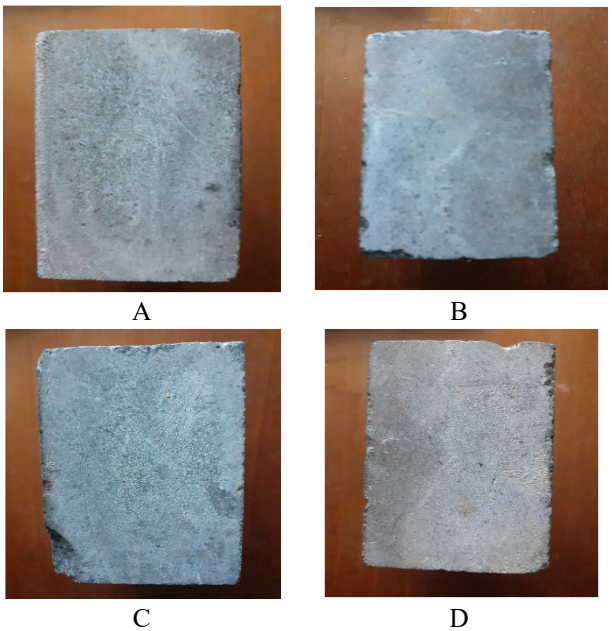

Figure 1. Surface aspect of specimens investigated at early-age
A – variant 1; B – variant 2; C – variant 3; D – variant 4.

The microstructural appearance of the four concrete specimen variants is shown in Figure 2.

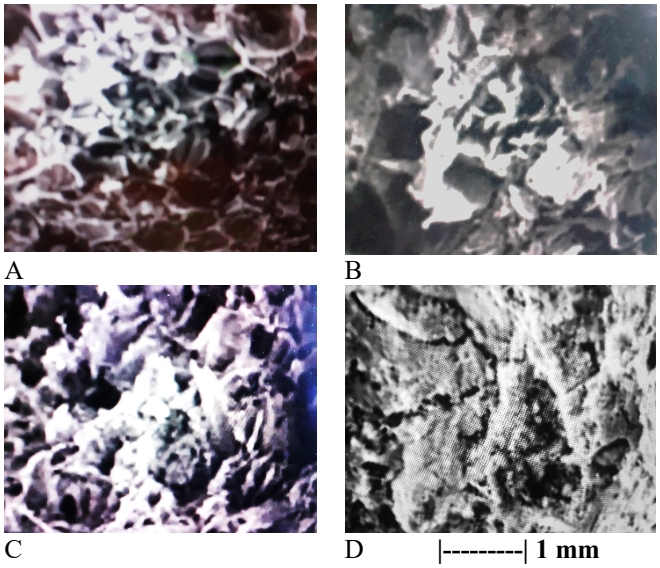

Figure 2. SEM images of concrete specimens at untimely carbonation
A – variant 1; B – variant 2; C – variant 3; D – variant 4.

Images presented in Figure 2 indicate the rather advanced compactness of the microstructure of concrete specimens characterized by the capture and storage of CO_2. The porousness of specimens is slightly decreasing from version A to version D, simultaneously with the increase of the $NaHCO_3$ proportion in the material mixture (increasing from 10 to 23 %). The pore size of the experimental concrete specimens is very low especially in the experimental versions made with higher amounts of $NaHCO_3$ (versions C and D).

3.2 Comments

Recent research has been begun around the world for the sequestration and definitive storage of carbon dioxide as carbonates, aiming to reduce these emissions into the atmosphere. C-S-H, created by hydrating the calcium silicate from Portland cement as well as transitional calcium hydroxide as nanoparticles, penetrate the porous spaces of undried cement, crystallizing. Since the principal objective of research is growing the concrete resistance at untimely carbonation, the option of introducing $NaHCO_3$ into the cementitious mix, replacing within the limits of 10-23 % of the initial quantity of cement, was unanimously accepted by researchers based on previous experimental results. It was found that, under the influence the $NaHCO_3$, the $Ca(OH)_2$ ions available at the direct contact between water and clinker facilitate turning the $Ca(OH)_2$ particles

into an amorphous material, acting as a nucleating agent for the formation of calcium silicate hydrate.

Other process-specific turning creates a composite based on calcium carbonate and C-S-H phase, with the ability to accelerate the hydration process of the clinker. In this way, the method of untimely binding of carbon dioxide in the fresh concrete ensures both the storage of carbon dioxide into the fresh concrete through its carbonation, and growing the untimely mechanical strength of concrete.

This procedure has not yet been finalized in technological terms. Its importance lies in the possibility of manufacturing without CO_2 emissions and using traditional Portland cement as the basic binder for construction concrete in the actual world ecological crisis.

The building materials industry is one of the industrial sectors where reducing the carbon footprint is difficult to achieve only by using alternative cementitious materials or renewable energy sources. According to reference [16], carbon capture and storage currently represents the only suitable option in this sector to avoid CO_2 release into the atmosphere.

The outcomes obtained as a result of the experiment described above clearly showed the consequence of $NaHCO_3$ supplementary added on the physio-thermal features, on the one hand, and respectively, on mechanical properties of the analyzed concrete specimens.

Compared to the physio-thermal characteristics of the reference concrete, the apparent denseness values of all four versions (A-D) are higher (within the limits of 2198-2231 $kg \cdot m^{-3}$, compared to 2178 $kg \cdot m^{-3}$), while the thermal conductance falls within the limits of 0.415-0.434 $W \cdot m^{-1} \cdot K^{-1}$, generally slightly lower than the 0.432 $W \cdot m^{-1} \cdot K^{-1}$ corresponding to the reference concrete (except for version D whose value is almost similar). Instead, the increase in the compression and flexural strength values of concretes manufactured using the new technique is obvious after 28 curing days, especially in the case of version D. As noted above, the strength values reached at early-age, identified in the determinations carried out after only 6 hours, are remarkable. Of the four versions tested, the optimal version was considered to be the one in which the amount of $NaHCO_3$ added as a partial substitute for Portland cement was 23 % (i.e. version D).

The maximum percentages of increasing the compression and flexural strength values obtained in this experiment compared to those corresponding to the reference concrete were calculated at 12.9 and 11.0 %, respectively. According to the literature [8], accelerating C-S-H carbonation by sequestering CO_2 into concrete can favour an increase of its strength within the limits of 2-18 %.

By comparison, tests previously carried out by some authors of the current work, under conditions where the maximum proportion of $NaHCO_3$ added to the concrete preparation mixture based on CO_2 storage through C-S-H carbonation was 21 % [17], led to maximum mechanical strength performances much lower than those obtained in the current work (compression strength of 43.4 MPa and, respectively, flexural strength of 11.9 MPa), with the observation that after only 8 hours high resistance values were identified, close to the maximum values corresponding to the 28-day curing process.

4. CONCLUSION

Reducing the carbon footprint, especially in industrial sectors, is a very important objective facing specialists worldwide. The cement industry, which contributes about 7 % to total greenhouse gas emissions, has a very difficult task in diminishing the carbon footprint of Portland cement. This cannot be achieved only by using alternative cementitious materials or renewable energy sources. The capture and permanent storage of CO_2 in traditional construction materials (cement, concrete) seems to be the only adequate option for protecting the Earth's atmosphere against CO_2 emissions. It has been experimentally found that the optimal method for sequestering CO_2 in concrete is the use of sodium bicarbonate ($NaHCO_3$) in the starting material mixture due to the untimely carbonation of C-S-H, the principal phase of the ordinary cement. Another effect of C-S-H carbonation is the significant improvement of the mechanical strength of concrete and the acceleration of reaching high strength values in a very short time interval. The proportion of $NaHCO_3$ added to the initial material mixture was included in the range of 10-23 %. Its optimal value was experimentally determined at 23 %, contributing both to the storage of CO_2 in concrete and to the increase of compression strength by 12.9 % and flexural strength by 11 % compared to strength values corresponding to the reference concrete.

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