VOL. 12, NO. 8, APRIL 2017 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

www.arpnjournals.com

EFFECT OF CARBON DIOXIDE ON THE DURABILITY OF CLASS-G API CEMENTS AT RESERVOIR CONDITIONS

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ABSTRACT

In the reservoirs it is known that in addition to formation water, oil and natural gas, there is carbon dioxide (CO_2) dissolved in high concentrations, whose effect on the durability of the cement placed behind the coater is unknown and constitutes the object of The present investigation. This research considered in the design and implementation of two experiments in the laboratory that allowed evaluating the effect of the CO_2 on the durability of the cement. Both experiments were carried out on samples of Class-G API cement which were exposed to different conditions of high pressure and temperature inside one litre capacity Parr stainless steel reactors in environments of supercritical CO_2 super saturation, The characteristics of the test specimens were then evaluated through tests such as VPU (ultrasonic pulse velocity), XRD (X-ray diffraction), compression strength, among others. The results obtained allowed to conclude that in the studied conditions the carbonation and retrogression phenomena occur simultaneously, which affect in a different way the durability of the cement. Retrogression involves micro fracture of the cement which results in loss of compressive strength and in the creation of flow channels, whereas carbonation initially implies increased compressive strength followed by loss of resistance due to the dissolution of calcium of the cement. The net loss of compressive strength is close to 50%; this results in loss of cement durability, which is one of the possible causes of water channelling in fields rich in carbon dioxide.

Keywords: durability, cement API G-class, adhesion, carbon dioxide CO_2, carbonation, VPU ultrasonic pulse rate, XRD x-ray diffraction, retrogression, compressive strength, hydraulic insulation.

INTRODUCTION

The need to improve the productivity and strategic development of the hydrocarbon sector, has led the operating companies to implement new methodologies that indicate the factors with greater incidence in the production.

It is known that in the deposits there are high concentrations of CO_2 at high pressure and temperature, dissolved in water rich in chlorides, bicarbonates, calcium and sodium, which alter the durability of the cement placed behind the coater and cause important physicochemical changes in the Itself, altering its resistance to compression and allowing the increase of water cut as production fluid.

In oil wells, cementation quality is evaluated indirectly through cementation profiles (CBL-VDL), which measure the response of cement to ultrasound, but ignore the chemical processes and mineralogical changes that can occur in the Cement and which affect its durability.

The state of the cementation is indispensable to selectively produce the fluids of the subsoil. Defective cementation can cause the invasion of the hydrocarbon-producing areas with water from underground levels. The aim of the laboratory studies is to evaluate the durability of cements due to the corrosive effect of CO_2, under high pressure and temperature conditions, simulating the conditions under which cements are exposed in the subsoil.

In the oil industry, specialized laboratory equipment is very expensive; the research remains very small, so that manufacturing them with state-of-the-art devices is a way to improve the endowment of the laboratories of the universities that are dedicated to this type of investigation.

METHODOLOGY

The project was divided in two experimental phases that had as common objective to evaluate the deterioration of the cement exposed to high concentrations of CO_2 to reservoir conditions.

The initial phase consisted of an accelerated carbonation experiment (experiment-1) in which twenty-eight (28) cement samples were exposed to the aggressive action of CO_2 over a wide range of pressure and temperature; The results obtained in this experiment allowed us to design the second phase of the project, which consisted of another accelerated carbonation experiment (experiment-2), in which the conditions were adjusted to investigate in depth the phenomenon of retrogression.

Experiment-1

The carbonation process is a very slow process; For which an accelerated carbonation process was implemented, in which cement was exposed to conditions of over saturation of CO_2, pressure and temperature determined, in stainless steel reactors type Parr of a liter of capacity. ECOPETROL S.A. Delivered 28 cylindrical specimens (14 large and 14 small) of CLASE-G API cement of known chemical composition, normally used in well-cementing works in the Caguán-Putumayo Basin.

These specimens were subjected to the accelerated carbonation process for sixty (60) days within the reactors with the following conditions: high salt concentration, CO_2 super saturation, reservoir-like

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pressures and temperatures and movement that simulate the dynamic flow conditions of the reservoir. Four samples were taken during the period of the experiment: one sampling every 15 days. The following table shows the detailed conditions.

Table-1. Conditions of aggression. experiment-1.

Reactor	**Pressure** (psi)	**Temperature** (°F)
R1	2000	180
R2	2500	200
R3	3000	220
R4	3500	240

Reactor conditions are considered more aggressive the higher the solubility of CO2 in the prepared brine. In the conditions proposed for the present experiment the solubility increases as a function of temperature and pressure. Therefore, it can be said that the reactors exposed to higher pressure and temperature are the reactors with the most aggressive conditions.

Experiment-2

The results obtained in experiment-1 allowed designing this second phase of the project, in which conditions were adjusted to investigate in depth the phenomenon of retrogression.

The accelerated carbonation process is evident in all specimens exposed to the action of CO2; to determine if the predominant process is carbonation or retrogression, a sensitivity analysis of the temperature parameter was carried out and it was observed that, in the samples exposed to temperatures higher than 230 ° F, the predominant process is retrogression.

Sixty 60 cement / cement ratio samples with a water / cement ratio of 0.441, 1.5 in nominal diameter and 3 in length, of known chemical composition, strictly following the API-10 standard [15] were made. We selected 24 specimens, the most suitable ones for the experimentation, the remaining specimens served as "white" specimens to carry out measurements of resistance to compression and porosity.

The specimens were distributed in 6 Parr type reactors. As shown in Table-2, larger pressure and temperature ranges were simulated to allow a clearer observation of the effect of temperature on the alteration of the cement.

Table-2. Conditions of aggression. experiment-2.

Reactor	**Pressure** (psi)	**Temperature** (°F)
R1	2000	205
	2200	205
R2	2400	225
R3	2600	235
R4	2800	255
	3000	255

The cement samples were exposed for 65 days by the action of a brine rich in sodium chloride (10'000ppm analytical grade NaCl). This composition did not attempt to simulate the subsoil conditions, but was formulated based on the collection of information, because the reference studies used this concentration of brine.

Tests performed

Phenolphthalein dissolution pH indicator. depth of carbonation. x-ray diffraction (XRD). Mineralogy of carbonates. Helium intrusion porosimetry and porosimetry method of saturation. Porosity. Ultrasonic pulse measurement. Travel time.

Destructive measurement of compressive strength. resistance to compression. measurement of weight. weight and density. calcium ion determination, atomic absorption method and EDTA method. concentration of calcium ion.

Results Experiment-1

Three different behaviors were identified throughout the experiment, each with different degrees of cement alteration. These three behaviors, called "frames of alteration", allowed analyzing the results in a more systematic way.

Front amendment-1 was presented between zero and thirty days of exposure. There is calcium leaching from the paste to the external medium, simultaneously the calcium of the brine ($CaCO_3$) precipitates on the surface of the test pieces and within the pore network at an average depth of 1.5 millimeters.

Front of change-2 was presented between the thirty and fifty days of exposure. The precipitation rate of $CaCO_3$ is much higher than the rate of calcium leaching. The density of the cement increases and the porosity decreases drastically.

Front of change-3 was presented between fifty and sixty days of exposure. There is simultaneously calcium leaching from the pulp, precipitation of $CaCO_3$ and dissolution of the already precipitated $CaCO_3$.

Visual Alterations: Exposure of cement specimens to the aggressive conditions mentioned produced very noticeable changes in their external surface, including their coloration and the presence of precipitates of different color and texture. Photo-1 shows the precipitate of one of the test specimens exposed to the action of CO_2.

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ARPN Journal of Engineering and Applied Sciences

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Photo-1. $CaCO_3$ precipitated in concrete specimens.

On the other hand, cement specimens exposed to temperatures higher than the retrogression temperature (> 230 ° F) showed slight micro fracture, as shown in photo 2. It should be noted that the initial state of the specimens shows initial micro fracture, and it is possible that most of the fractures were created during the setting process, prior to experimentation.

Photo-2. Micro fracture of cement. initial state.

It was observed for all cases that micro fractures facilitated the diffusion of CO_2 through the pore network, as shown in photo-3, which shows the micro fracture detected with the phenolphthalein test.

Photo-3. Micro fracture detected with phenolphthalein.

Weight Alterations: In general, weight gain was observed in all cement samples evaluated, as a result of the carbonation phenomenon.

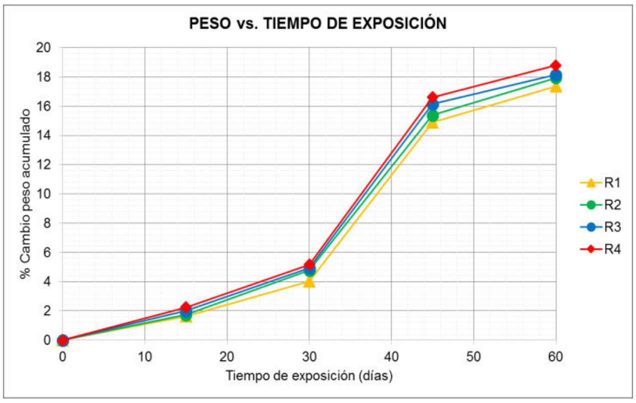

Figure-1. Weight change vs. exposure time.

Amendment front-1: The net weight gain of the cement samples was 5%.

Front of change-2: The net gain of weight was 15%. Due to the active carbonation, the weight increased more rapidly and the porosity decreased because of the precipitation of $CaCO_3$.

Amendment front-3: The increase in weight of the test specimens was attenuated. The precipitation rate of $CaCO_3$ was much higher than the rate of calcium leaching; However, $CaCO_3$ solution was presented in the same magnitude, governed by the bicarbonation process. For this reason, the density increased very slightly.

Ultrasonic travel time: $CaCO_3$ precipitated in the pore network and in the micro fracture network, generally affected the density of cement samples, and thus other properties such as transit time and compressive strength.

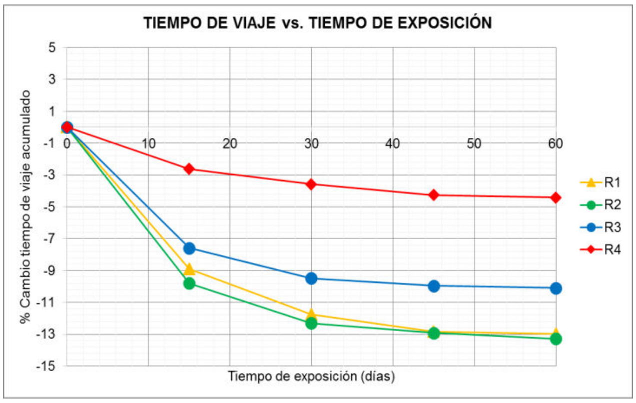

Figure-2. Alteration of travel time vs. exposure time.

It was observed that the travel time decreased as a function of the exposure time; During the AMENDMENT FRONT-1 travel time decreased with a slope ranging from 15% (> 240 ° F) to 66% (<220 ° F); After 30 days, the travel time was attenuated and decreased with a slope of 4% for all reactors (FRAMES OF AMENDMENTS 2 and 3). This is clearly related to the increase in density of the specimens; the sound finds a denser medium and travels at greater speed (less travel time).

Since ultrasonic waves travel preferentially through closed fractures, which oppose less resistance, the

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results suggest that the micro cracks created by the high temperatures were filled and sealed by the precipitated $CaCO_3$. However, although in the specimens that suffered retrogression, travel time decreased (higher density), rigidity had already been lost and the compressive strength was already affected. Therefore it was not possible to directly relate the decrease in travel time with the loss of compressive strength, as originally intended.

Depth of carbonation: The entrance of the brine to the poral network and to the network of micro fractures of the cement is governed by the diffusion behavior; it can be estimated that the thickness of the carbonate layer is proportional to the square root of time, according to Fick's law.

The depth of carbonation for each of the analyzed samples, obtained by the phenolphthalein technique, allowed us to calculate the value of the constant K or carbonation constant, which can be used to predict the depth of carbonation for different periods of time at Simulated conditions in the laboratory, and calculate the time at which the cement behind the coater is altered, causing loss of hydraulic insulation.

$$x = K\sqrt{t}$$

Equation 1. Fick's law

x = carbonation depth (mm);
K = coefficient of carbonation (mm / año0.5);
t = time (year)

Carbonation coefficients calculated as described in Table-3.

Table-3. Carbonation rates calculados.Experimento K-1.

Reactor	Carbonation coefficient	Statistical correlation (%)
R1	11.923	91.24
R2	15.877	91.93
R3	18.502	96.17
R4	25.362	98.13

The joint effect of retrogression and carbonation allowed the advance in the carbonation front to be faster because micro cracks created by retrogression increased the diffusivity of the saturated brine with CO2.

On the other hand, the carbonation process accelerated as a function of temperature. Figure-4 shows the changes in the carbonation coefficient as a function of temperature.

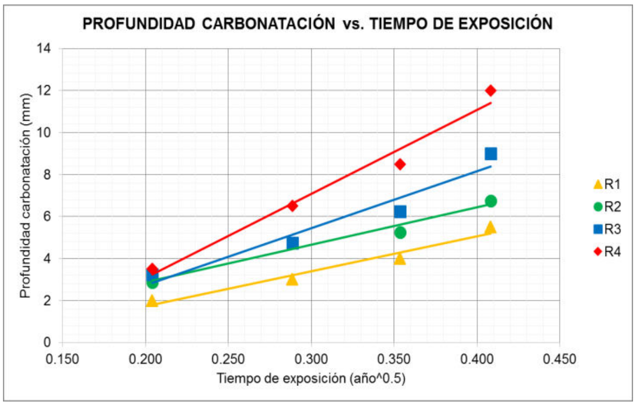

Figure-3. Depth of carbonation vs time of exposure.

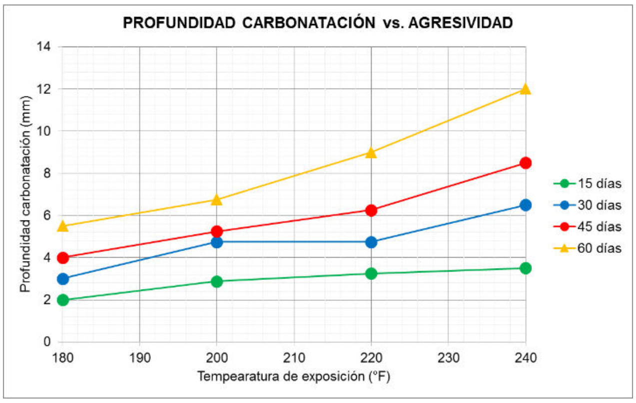

Figure-4. Coefficient carbonation vs calculated aggression.

X-ray diffraction: the CO_2 attack occurred preferably on the portlandite, being observed the generation of calcite, aragonite and vaterita. The high amount of calcium initially present in the brine allowed a more active precipitation of $CaCO_3$ which produced apparently favorable alterations in the cement structure. However, as will be seen below, the results of compressive strength showed that the precipitation of $CaCO_3$ was favorable only for the cement exposed to temperatures below 220 ° F.

It is believed that the precipitation rate $CaCO_3$ is proportional to the exposure temperature, which is verified with the carbonation depth data through the phenolphthalein test, observing that the higher the temperature the greater the invasion of the carbonation front.

Figures 5 and 6 present the diffract grams of specimens 11-A and 14-A, exposed to 240 ° F, 3500psi, and exposure time of 15 and 60 days respectively.

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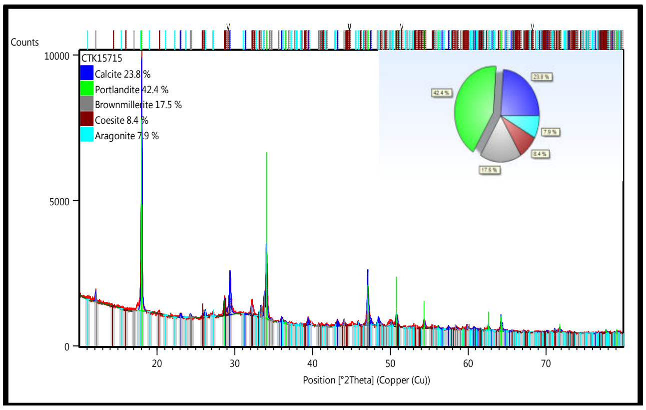

Figure-5. Diffract gram. sample 11A (15 day exposure; reactor-4).

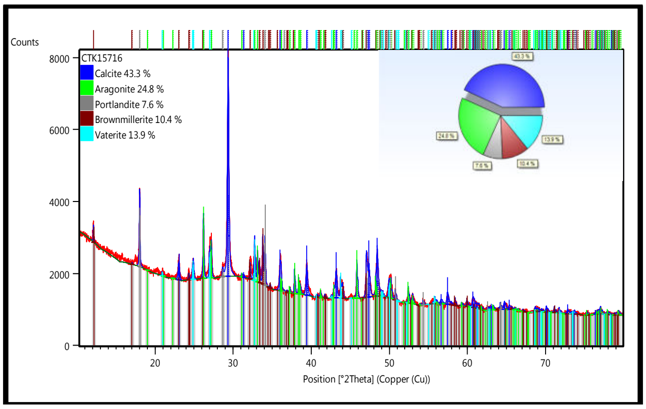

Figure-6. Diffractogram. sample 14A (60 day exposure; reactor-4).

Compressive strength: High compressive strength of cement is synonymous with good hydraulic insulation between the producing area and the water zone. [2. 3]

For the present experiment, samples 1A, 2A and 13 were randomly used as a reference when determining changes in compressive strength, which had average compression strength of 3500psi. Figure-6 shows the change in the compressive strength of the cement samples as a function of the exposure time.

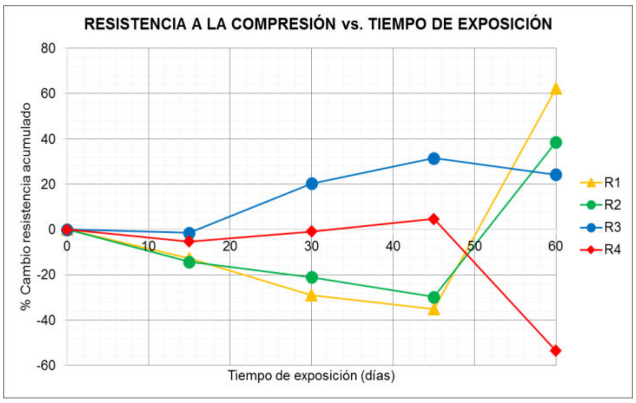

Figure-7. Compressive strength vs. exposure time.

Amendment front-1: Cement specimens exposed to temperatures higher than 220 ° F suffered from intense micro fracture which was counteracted by the active precipitation of $CaCO_3$, this precipitation registered a very slight increase in the compressive strength. On the contrary, the specimens that were exposed to a temperature lower than 220 ° F only suffered the carbonation phenomenon. Their resistance decreased as a result of the leaching of paste material about 10%.

Changing fronts 2-3: At temperatures above 220 ° F, fracturing became more intense, $CaCO_3$ precipitation created sealed fractures but failed to counter the loss of strength and stiffness suffered by the sample due to micro fracture, thus registering a decrease in the resistance to compression. At temperatures below 220 ° F, the active precipitation of $CaCO_3$ increased the compressive strength of the cement; As a consequence, cement samples increased up to 60% compared to their initial value.

At the end of the experiment the cement lost, in the most aggressive conditions (240 ° F), compressive strength with a slope close to 70%, and at the end of the experiment, a 50% reduction of the resistance with respect to the value of initial.

Experiment-2.

Two (2) different behaviors were identified throughout the experiment, each with different degrees of cement alteration. Which are denominated like "front of alteration".

The front of change-1: It was distinguished between 0 and 15 days of exposure and is characterized by loss or leaching of calcium from the paste. The $CaCO_3$ precipitation rate was very low, because the brine is low in calcium.

The front of change-2: It was distinguished after the 15 days of exposure, and is characterized by the high precipitation rate of $CaCO_3$ that exceeds the calcium leaching. The precipitation rate of $CaCO_3$ was controlled by aggressive conditions.

Visual alterations: The accelerated carbonation process caused $CaCO_3$ precipitation on the surface of the cement samples, as shown in photo-4.

Photo-4. $CaCO_3$ precipitated in cement samples.

The $CaCO_3$ precipitated on the surface of each sample was collected and weighed during each sampling;

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it was evaluated in the Water Laboratory of the Surcolombian University (LAUS). During the carbonating front-1 the highest $CaCO_3$ precipitation was recorded on the outer surface of the cement samples, due to the carbonation.

After 15 days of exposure (alteration front-2) the $CaCO_3$ present on the surface of the cement samples decreased by an average of 20% as a function of time.

On the other hand, the cement specimens subjected to the retrogression phenomenon suffered from a strong fracturing, as evidenced in photos-5.

Photo-5. Micro fracture of cement by retrogression.

In general it was observed that samples exposed to temperatures higher than 220 ° F suffered from intense fracturing. The fractures created by retrogression, facilitated the failure of the samples during the tests of resistance to compression. As shown in photo-6, the cement has preferential planes of failure.

Photo-6. Measurement of resistance to compression.

Weight alterations: Weight behavior is different from that observed during the first 15 days of experiment-1. Because the brine is low in calcium, the sense of precipitation of the different materials was reversed. Figure-7 shows the percent change in weight of the test specimens as a function of the exposure time.

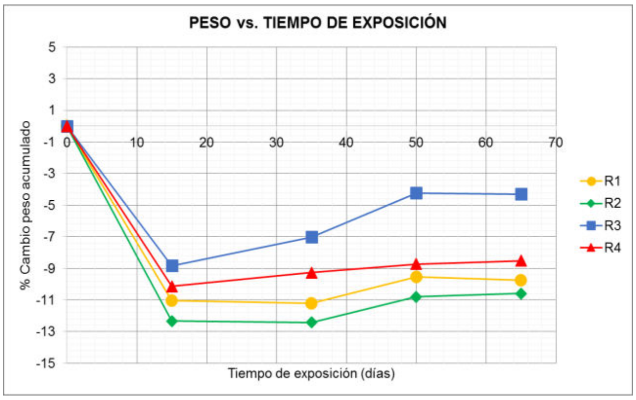

Figure-8. Weight change vs. exposure time.

Amendment front-1: Weight loss was observed in all cement samples evaluated, due to the calcium leaching of the paste; the weight decreased with a slope of 70%.

Front of change-2: The weight of the samples increased between 1% and 4%. The increase in weight evidenced the precipitation of $CaCO_3$ in the poral network. However, at the end of the experiment, the net weight change was -10%, compared to the initial weight; this suggests loss of cement durability.

It is anticipated that at longer exposure times, when the calcium of the pulp is exhausted, the overall density of the specimen will decrease drastically, due to the leaching of previously precipitated $CaCO_3$; this process is known as bicarbonatación, and is the main cause of the deterioration of the cement.

Porosity alterations: Porosity behavior is similar to that observed in experiment-1: it increased due to the calcium leaching of the paste and decreased as a consequence of the precipitation of $CaCO_3$. Figure 8 shows the percent change in porosity of the test specimens as a function of the exposure time.

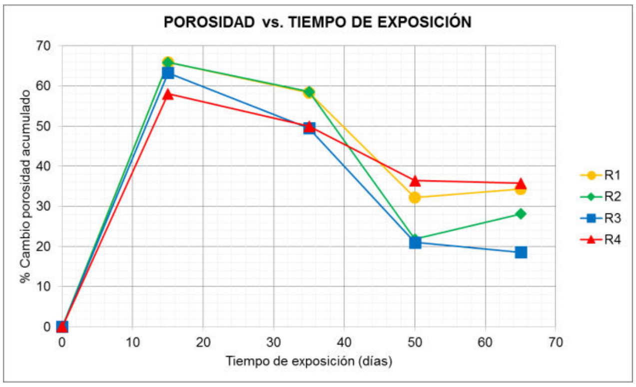

Figure-9. Alteration of porosity vs. time of exposure.

Alteration front-1: Porosity increased with a slope of 70% due to the calcium leaching of the paste.

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Front of alteration-2: Precipitation of CaCO3 decreased porosity between 30 and 50%. In spite of this precipitation, the net variation of the porosity was 30% above its initial value.

It is anticipated that in times of longer exposure, when the calcium of the paste is exhausted, the porosity of the cement specimen increases drastically, due to the bicarbonation; this would imply loss of compressive strength and creation of flow ducts that decrease hydraulic insulation.

Ultrasonic travel time: The decrease in weight of cement specimens caused an increase in travel time during the Change front-1. After the 15 days of exposure (Front of change-2), as a consequence of carbonation, the wave found a more dense path through which to observe a decrease in travel time.

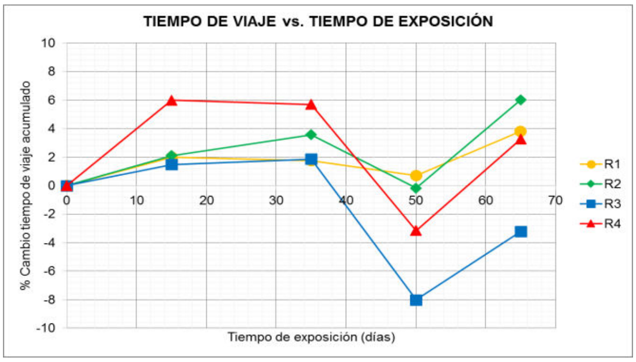

Figure-10. Alteration of travel time vs. exposure time.

The aggressiveness of the system shows that the travel time was affected more in conditions of higher temperature. At higher temperature, the precipitated $CaCO_3$ was higher, and the ultrasound wave found a means by which to transit more easily.

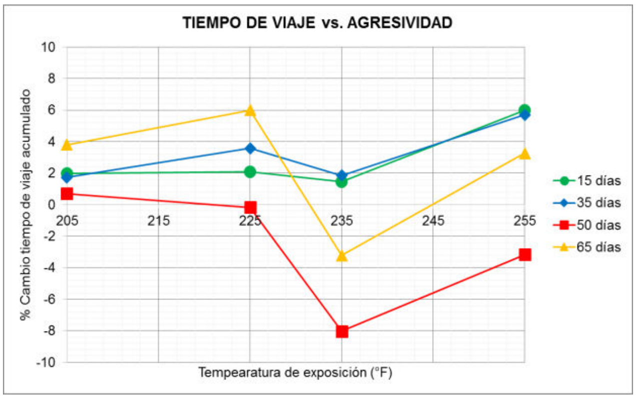

Figure-11. Alteration of travel time vs. aggressiveness.

Carbonatation depth: In general, the carbonation depth observed in the present experiment is greater than that observed in the carbonation experiment of phase-1. The calculated carbonation coefficients are described in Table-4.

Figure-11 shows the average carbonation depth for the four conditions studied, from which the carbonation coefficient K was calculated.

Table-4. Calculated K-carbonation coefficients Experiment-2.

Reactor	Coefficient of carbonation	Statistical correlation (%)
R1	20.854	85.79
R2	23.894	87.71
R3	40.202	86.40
R4	27.913	94.59

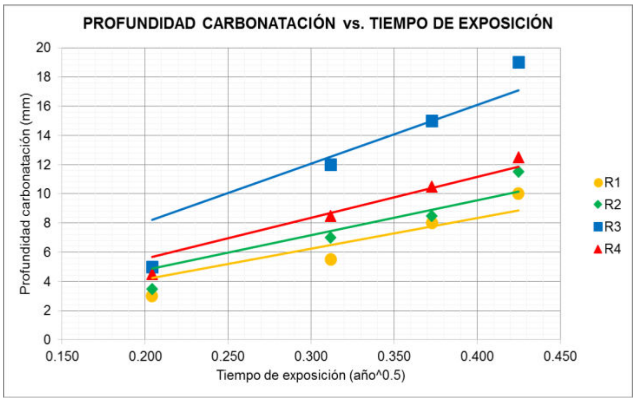

Figure-12. Depth of carbonation vs. time of exposure.

For the more aggressive conditions, the carbonation was preferentially developed around the fractures, the advance of the carbonation front decreased. The micro fracture network allowed the precipitation of $CaCO_3$ to be more active, creating sealed or closed fractures, which although they meant increased density, did not necessarily imply an increase in the compressive strength.

Compression resistance: Resistance behavior was similar to that observed in Experiment-1. Figure-12 illustrates the alteration of the compressive strength of the cement as a function of the exposure time, for different aggressive conditions.

The most aggressive conditions (Reactor-4) presented initial resistance increase, followed by a drastic fall of the same, product of the network of micro fractures created by the retrogression. The conditions of low aggressiveness presented resistance drop during the first 15 days; then they tried to increase their resistance, as in experiment-1. However, for these experimental conditions, because the calcium content of the brine is low, the rate of precipitation of calcite was not high enough for the resistance to rise above the initial value.

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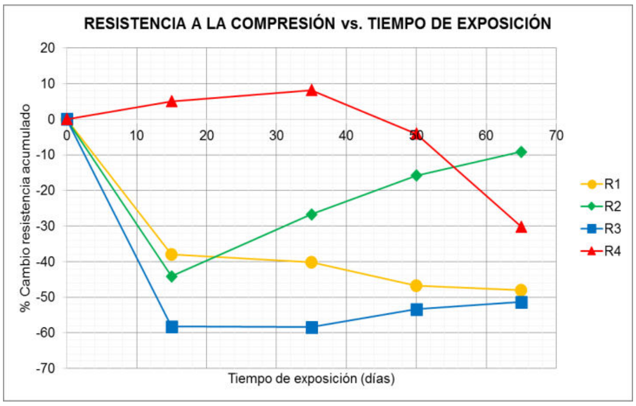

Figure-13. Compression resistance vs. exposure time.

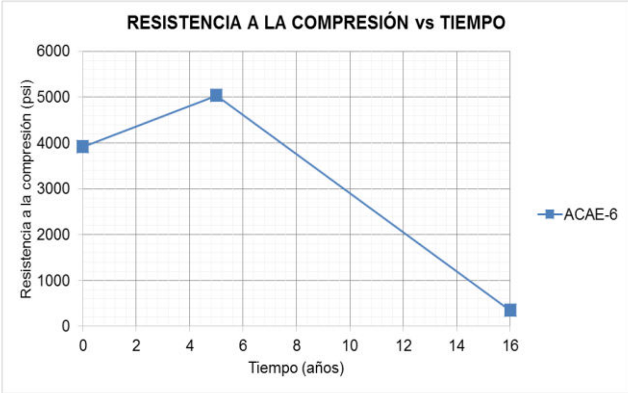

Figure-14. Resistance to compression vs. exposure time. Well Acae-6.

The loss of net compressive strength for high temperature conditions was 30%. For the low and medium temperature conditions, the initial resistance loss was 60%, and recovered as a function of time; at the end of the experiment, between 10 and 50% resistance to compression was lost. It is observed that at the end of the experiment that both carbonation and retrogression caused loss of compressive strength of the samples.

Data obtained in laboratory vs. data obtained in the field: In order to extrapolate the results obtained to the field conditions, an attempt was made to relate the loss of resistance to compression seen in the laboratory, with the loss of the compressive strength calculated by the Cementing profiles.

According to the inventory of cementation profiles registered in the study area, the ACAE-6 well is the only well in the area of interest that has different cementing profiles CBL-VDL at the same depth and different time periods.

The amplitude values read from the CBL profiles were converted to compressive strength values using the Schlumberger cement bond interpretation chart. The values of compressive strength allowed interpreting the state of the cementation and the hydraulic insulation between the zone of interest and the water zone.

Figure-13 shows the alteration of the average compressive strength as a function of time, obtained through the Schlumberger chart, for the interval 10550 to 10600 feet.

It was observed that initially the resistance to compression increased, and after 4 years it decreased drastically. The behavior of the compressive strength alteration curve is similar to that observed in cement samples exposed to temperatures higher than the retrogression temperature.

In terms of hydraulic insulation, for the specific case of ACAE-6 well cement (7in outer diameter (OD) and specific weight 29lb / ft.), the recommended cut-off of the compressive strength is Of 685 psi, which means that any cement with a strength value below this value is considered unfit and must be replaced or remedied.

CONCLUSIONS

The cement of the test pieces suffers from the carbonation process when it is exposed to the action of the CO_2 in high concentrations and in the supercritical state.

The velocity of the carbonation suffered by the cement of the specimens exposed to the action of the CO_2 is governed by the law of diffusion of Fick. The calculated coefficients allow predicting the depth of carbonation of the cement in the subsoil.

The reaction between the CO_2-saturated brine and the calcium contained in the cement specimens produced an irregular carbonation front that extended up to 15mm from the outer edge of the sample towards its center. The carbonation front is lighter in color and identified as colorless through the phenolphthalein test.

The DRX tests on the samples exposed to the most aggressive conditions (3500psi, 240 ° F) showed that the mineral phase that precipitates during the carbonation process corresponds to calcium carbonate ($CaCO_3$) in the form of calcite mainly and of aragonite in minor proportion.

The first alteration that suffered the cement exposed to the action of the CO_2 in high concentrations and in supercritical state was due to the leaching of calcium. Consequently, the porosity increased by an average of 60% and the weight decreased by about 10%.

In the EDTA analysis carried out on the brines to which the cement was exposed, evidence of calcium leaching from the paste.

In calcium-rich brines, the cement of the test specimens was exclusively affected by the carbonation process and due to the precipitation of $CaCO_3$ in the pore network and in the network of fractures the resistance to compression was increased.

In calcium-poor brines, the cement underwent the bicarbonation or dissolution of $CaCO_3$ previously precipitated in the pore network, consequently the compressive strength decreased.

The micro-fractures generated by retrogression facilitated the diffusion of CO_2. As a consequence, the carbonation depth was higher in cement samples exposed to higher temperatures.

The cement of the specimens that suffered retrogression lost about 55% of their resistance after 60 days, whereas in the cement of the specimens exposed to temperatures below 220 ° F the resistance increased because of the carbonation phenomenon.

The electronic automation of the Ultrasonic Travel Time test allowed reducing the error in the results up to 10% and the execution time in 2 min per sample.

It was not possible to relate the travel time of a compressional wave to the compressive strength, due to the small number of samples that were exposed to the experimental conditions.

In the ACAE-6 well the compressive strength decreased as a function of time, about 75%. The ACAE-6 is the only well that has CBL-VDL records taken over a 21-year span.

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