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Construction of synthetic carbonate plugs: A review and some recent developments

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Abstract. Plugs are cylindrical rocks with known dimensions that are extracted typically from reservoir formations with representative mineralogical compounds, petrophysical properties and oilfield fluids. They are used in the laboratory to understand the behaviour of oil in reservoirs. One of their applications is to study the screening of chemicals, such as surfactants and polymers, for enhanced oil recovery research before being applied in the reservoir. Many of Brazil's pre-salt basins are located in ultra-deep waters, and the high heterogeneities of its offshore carbonate reservoirs make the extraction of representative rock samples difficult, risky and expensive. The literature reports the construction of synthetic plug samples that reproduce rocks as an alternative and viable solution for this issue. However, there is a lack of publications that focus on the construction of representative carbonate plugs that considers both the mineralogical composition and petrophysics properties, such as porosity and permeability. In this work, the construction of synthetic plugs is studied, using a combination of published methodologies to achieve an alternative construction of synthetic carbonate plugs for laboratory scale studies. Using a procedure based on the use of pulverized rock matrices with known particle sizes, uniaxial compaction, and probable $CaCO_3$ solubility control by changing temperature and pH, it was possible to obtain synthetic carbonate plugs with a similar mineralogy to the natural carbonate reservoir. However, further studies are necessary to obtain more controlled petrophysical properties of such samples.

1 Introduction

By definition, core plugs are rock samples extracted from a reservoir with specific dimensions, about 25.4–38.1 mm in diameter and a length 1–1.5 times greater than the diameter [1]. This sample provides a relative macro scale version of the oil and gas reservoir, due to its representative fluid content, mineralogical and petrophysical properties [2].

The sample plugs are used for basic and special core analysis. The basic information obtained includes porosity and permeability, and is used to provide input data for reservoir characterization in areas like reservoir engineering, geology and petrophysics. Specific tests allow the evaluation of the effect of reservoir production performance, such as fluid displacement on enhanced oil recovery studies [3, 4].

Plugs are formed by carbonates or sandstones, which are the main conforming types of rock in oil and gas exploration areas [5]. These two types of rocks have significant differences. The first is the sediment production location, and the second is the higher chemical reactivity of the carbonate mineral. The latter results in heterogeneous reservoirs due to chemical cementation and fractures generated by lower resistance to compaction [6]. Due to the high natural depths of hydrocarbon carbonate reservoirs, such as the Brazilian Presalt that is 300 km from the coast to 5000 m of depth, it is difficult, risky and expensive to obtain natural sampling plugs [6, 7].

In cases where reservoir samples are not available or are quite expensive to obtain, alternatives such as commercial rock plugs extracted from mines, or constructed in the laboratory (synthetic), are available [8]. The latter option, having variable and known characteristics, such as mineralogy and petrophysics, may represent a closer approximation to the reservoir samples.

Initially, this paper focuses on a literature review that describes the lack of studies for the construction of synthetic carbonate plugs. Next, it proposes an exploratory methodology to reproduce both physical (petrophysical) and chemical (mineralogy) rock characteristics, enabling the generation of promising studies to understand the

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behaviour of carbonates in oil and gas reservoirs, such as the Brazilian Presalt.

2 Review approach

A selection of papers was screened by using the following keywords: lithology (carbonate), rock sampling (plug or core) and sample feature (synthetic or artificial). Only papers focused on the use of such samples for the petroleum engineering area, and the ones where the construction methodology could be identified, were considered. In addition, some undergraduate and master's degree projects were analysed. In this way, 81 studies were included [8–88].

Analysis of the chronology of the studies, construction materials used and consolidation methodology for the plugs will be presented in the following sections.

2.1 Synthetic plug construction chronology

Work in this area has been ongoing for 54 years, starting in 1962. Figure 1 shows an increasing number of publications, from seven papers in the 1960's to 38 in the last decade, demonstrating the growing interest in this area of petroleum engineering.

2.2 Materials and contents used to construct synthetic plugs

Due to the relationship between the rock chemistry and the material used to construct synthetic plugs, it was important to identify the main materials and their contents which were used to construct such samples, independent of the approach used for their construction, such as uniaxial compression, cementing of materials, sintering and others.

In this way, from 18 of the 81 papers, it was possible to identify 10 different materials and theirs contents. Table 1 presents these materials, their content range and associated publications.

From Table 1, it is possible to identify the materials and content range from 50% to 100% of the synthetic plugs. Note that from these 10 materials, 6 correspond to sand matrices, and the rest include crushed formation rock, $CaCO_3$, bentonites and kaolinites. Most of these selected studies were therefore based on the construction of sandstone plugs. There are two likely reasons for this finding. The first is that constructing a porous media with a naturally granulated material, with known particle size, is fast and easy. The second is that because it is a porous media, it allows studies with no influence of chemical reactivity, due to the simple chemical composition of the sand (silica and oxygen) [31].

Regarding the approach for construction of carbonate plugs, from these 18 selected papers, it was possible to identify one that used a carbonate matrix material and its content [12]. Using calcite flour as its main component material (95% w/w), carbonate plugs were constructed for the application of electrical current to increase the flow rate of oil and water in a porous media. The lack of papers using mainly a carbonate matrix material demonstrates the need for research focusing on this mineralogical composition.

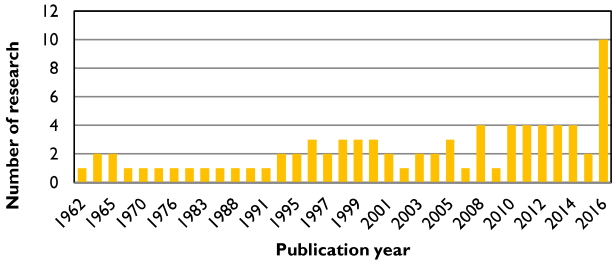

Fig. 1. Papers published about synthetic plug construction as a function of time.

Additional papers were identified that used a non-carbonate matrix material, such as sands, but used cementing procedures to allow the calcium carbonate precipitation. Previous literature evaluated a quartz sand (50% w/w) cemented in a medium favourable to precipitation of calcium carbonate to simulate a carbonate medium. The aim was to evaluate the geochemical interactions with a reactive fluid for carbon dioxide geological storage purposes [64]. The findings and discussion about these approaches will be detailed in Section 2.4.4.

According to reference [89], petrophysical properties of rocks depend on the particle size, amount of cementation and compaction. For this reason, the next analyses from this literature review will be focused on these parameters.

2.3 Particle size

It is important to mention the importance of the particle size distribution on porosity and permeability.

Particle size is defined as the size of each particle which compounds a porous media. Naturally, a rock is composed of different particle sizes, which generates a heterogeneous system. Empty spaces called pores are generated between these particles, and fluids, such as oil and gas, can be stored in these spaces in reservoir rock. The measurement of this empty space, commonly known as porosity, is defined by the relation between the volume of the empty space and the bulk volume of the sample (where pore volume is dependent on the volume of particle, and therefore also dependent on its size) and calculated using equation (1) [89, 90].

$$\phi(\%) = \frac{\text{Pore volume}}{\text{Bulk volume}} \times 100 = \frac{\text{Bulk volume} - \text{Solid volume}}{\text{Bulk volume}} \times 100. \quad (1)$$

In the case of an ideal system with an arrangement of uniform spheres and a fixed bulk volume, the porosity will be independent of the particle volume, and therefore of the particle size. But in a real system with particles of different sizes, smaller particles enter the empty spaces between the larger particles, thus decreasing the porosity. This demonstrates the importance of the particle size on the resulting porosity [90]. Figure 2 presents a schematic of three porous solids with the same dimension and composed by different particle sizes: (A) large uniform particle, (B) uniform smaller particle size and (C) different size

Table 1. Main minerals used to construct synthetic plugs.

Minerals	Constructed plugs	Content range (%)	References
Sand	9	70–98	[14, 18, 27, 31, 59, 66, 67, 71]
Silica sand	8	80–100	*i.e.*, [8, 10, 12, 15, 17]
Quartz sand	3	50–85	[50, 64, 85]
Carmel silica sand	2	95	*i.e.*, [12]
Crushed rock[a]	2	95–100	*i.e.*, [42]
Ottawa silica sand	1	90	[12]
Toyoura silica sand	1	98	[79]
$CaCO_3$ fluor	1	95	[12]
Bentonite	1	100	[79]
Kaolinite	1	100	[79]

[a]Lithology non-identified.

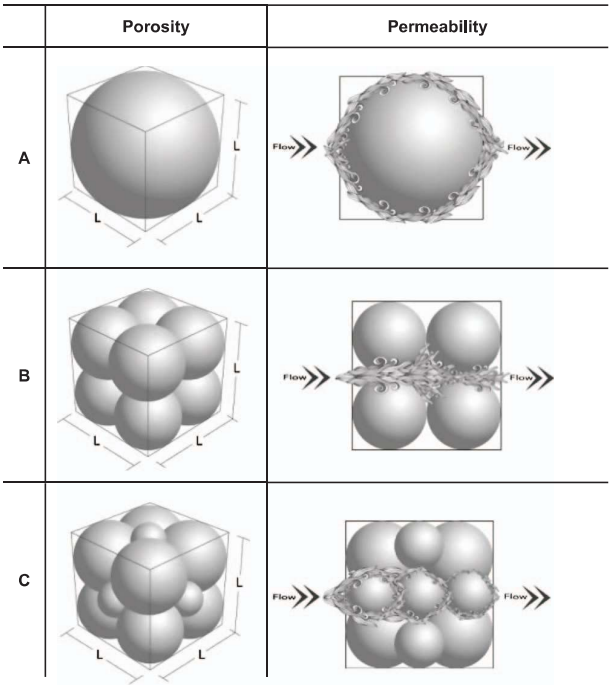

Fig. 2. Porosity and permeability due to (A) uniform particle of large size, (B) smaller uniforms particles and (C) different sized particles.

particles. For these cases, the porosity will be demonstrated based on equation (1). The bulk volume of the three solids with dimensions, L, was calculated.

$$\text{Bulk volume} = L \times L \times L = L^3. \tag{2}$$

Subsequently, it was necessary to calculate the volume occupied by the particles in each of the examples cited above. For this, it was necessary to employ the following volume sphere equations: (A) $L/2$ radius, (B) 8 spheres of $L/4$ radius and (C) 8 spheres of $L/4$ radius for larger particles, and 6 spheres of $L/6$ for smaller particles.

$$\text{Grain volume case A} = \frac{4}{3}\pi\left(\frac{L}{2}\right)^3 = \frac{1}{6}\pi L^3, \tag{3}$$

$$\text{Grain volume case B} = 8 \times \left(\frac{4}{3}\pi\left(\frac{L}{4}\right)^3\right) = \frac{1}{6}\pi L^3, \tag{4}$$

$$\text{Grain volume case C} = 8 \times \left(\frac{4}{3}\pi\left(\frac{L}{4}\right)^3\right) + 6 \times \left(\frac{4}{3}\pi\left(\frac{L}{6}\right)^3\right) = \frac{11}{54}\pi L^3. \tag{5}$$

Finally, using equation (1), the porosity of these volumes was calculated:

$$\phi(\%)\,\text{A} = \frac{L^3 - \frac{1}{6}\pi L^3}{L^3} \times 100 = \frac{6-\pi}{6} \times 100 = 47.6\%, \tag{6}$$

$$\phi(\%)\,\text{B} = \frac{L^3 - \frac{1}{6}\pi L^3}{L^3} \times 100 = \frac{6-\pi}{6} \times 100 = 47.6\%, \tag{7}$$

$$\phi(\%)\,\text{C} = \frac{L^3 - \frac{7}{24}\pi L^3}{L^3} \times 100 = \frac{54-11\pi}{54} \times 100 = 36.0\%. \tag{8}$$

According to the obtained results, it was shown that in ideal systems with uniform particles, as for cases A and B, the resulting porosity is exactly the same, corroborating the non-relation between particle size and porosity. Otherwise, in the real system, C, that contains particles of different sizes, the presence of smaller particles affects the porous space and decreases the porosity. In the same way, similar reductions for porosity are expected due to the influences

of compaction and cementation. This occurs because empty spaces will be filled by particles or cementitious materials, which decreases the percentage of pores in a solid sample. In addition, the ability of the oil stored in the pores to flow through a reservoir rock, known as permeability, has a direct relationship with the particle size, consolidation and cementation of the sample.

In cases where a solid possesses connected pores, the presence of smaller particles occupying the empty spaces, as well as high degrees of compaction and cementation, generally reduce the permeability. This can be corroborated by the mathematical equation by Darcy for the flow of a fluid in a porous medium, by studying the pressure gradients [89, 90].

$$\upsilon = \frac{q}{A_c} = -\frac{k}{\mu}\frac{\mathrm{d}p}{\mathrm{d}l}, \tag{9}$$

where:

υ = fluid velocity, cm/s,
q = flow rate, cm^3/s,
k = permeability of the porous rock, Darcy,
A_c = cross-sectional area of the rock, cm^2,
μ = viscosity of the fluid, cP,
l = length of the rock sample, cm,
$\frac{\mathrm{d}p}{\mathrm{d}l}$ = pressure gradient in the direction of the flow, atm/cm.

Figure 2 presents an interpretation of the flow of a fluid within three types of porous solids. From here, it is possible to demonstrate the application of the previous mathematical equation by Darcy. Flow restrictions caused by a decrease of the porous space due to the presence of particles of different sizes, compaction, or cementation result in a increase in the pressure gradients, and therefore lower permeabilities.

It is important to mention that from 81 articles, only 33 described particle sizes, allowing the identification of a range from 0.017 mm to 6 mm. To have a better view of the particle sizes used, the times in which these granulometries were used *versus* particle size was plotted. Figure 3 presents this particle distribution. It should be noted that the thickest and finest particles were used less. This could be explained by causing a weak consolidation of the sample for the former, and a substantial decrease in the porosities and permeabilities for the latter. However, fine particle sizes

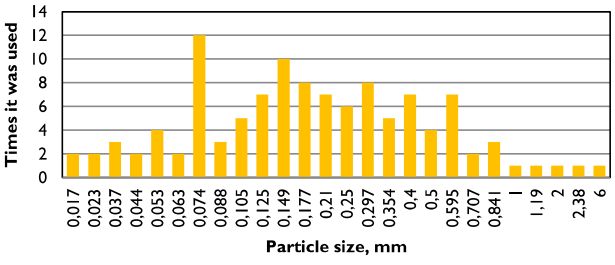

Fig. 3. Particle size based on the literature review to construct synthetic plugs.

Table 2. Particle size used to construct synthetic plugs.

Particle size [mm]	Reference
6	[82]
2.38	[84]
2	[84]
1.19	[84]
1	[13]
0.841	[10, 13, 19]
0.707	[10]
0.595	[10, 84, 86]
0.5	[82, 86]
0.4	[50, 52, 86]
0.354	[50, 52, 86]
0.297	[27, 50, 52, 84, 86]
0.25	[50, 86]
0.21	[41, 50, 51, 86]
0.177	[34, 50, 84, 86]
0.149	[17, 50, 55, 84, 86]
0.125	[21, 50, 55, 86]
0.105	[18, 55, 86]
0.088	[18, 55, 86]
0.074	[9, 15, 18, 25, 41, 84, 86]
0.063	[25]
0.053	[25, 27]
0.044	[25]
0.037	[12, 86]
0.023	[12, 86]
0.017	[86]

can improve the consolidation behaviour due to the particle agglomeration by compression [91]. In addition to this, a clear tendency to use particle from 0.841 mm to 0.037 mm sizes and close to 0.149 mm was identified.

Table 2 presents papers where these particle size materials were used to construct synthetic plugs. For example, in the work of [27], two materials with a particle size distribution centred around on diameters 0.053 and 0.297 mm, were cemented with epoxy resin to construct a porous medium to study the acoustic behaviour, in order to compare the permeability of synthetic sandstone samples.

2.4 Methodology for consolidations of synthetic plugs

The methodology used for consolidation of the plugs was categorized into five groups: *Group 1*: unconsolidated samples, *Group 2*: consolidated by compaction, *Group 3*: consolidated by bonding materials, *Group 4:* consolidated by material precipitation and *Group 5:* consolidated by materials sintering (Fig. 4). In a first analysis, it was found that consolidation using bonding materials is the most used consolidation method, corresponding to 32 studies. Of these, 29 studies correspond to the use of epoxy resins and 6 to Portland cement as a bonding material. Subsequently, other works correspond to compacted samples (21) consolidated

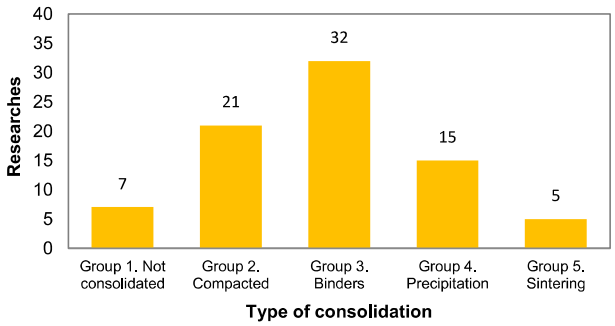

Fig. 4. Methodologies for synthetic plug consolidation.

by material precipitation (15), unconsolidated samples (7) and finally consolidated by sintering (5). Additionally, it was observed for all methods, drying or sintering processes set from room temperature to 190 °C.

2.4.1 Group 1: Unconsolidated samples

A rock plug sample may be unconsolidated, as with unconsolidated sand, depending on the oilfield properties as in the case of [50]. For this reason, after a particle size analysis of reservoir rock particles, the author mixed sands of sizes between 0.4 mm and 0.125 mm, and this was used to represent the reservoir zone of interest.

Another material conforming to unconsolidated plugs was found [82]. Here, packed glass beads of different sizes were used to study the chemical interaction of fluids within the porous media. The authors justified the control of porosities and permeabilities due to the use of glass beads and its physical standard characteristics. Based on the same packaging principle, the authors of [61] packed and sterilized at 165 °C for 3 h quartz sand of size 0.595–0.297 mm in a stainless steel tube to be used in microbially enhanced oil recovery flooding. The sterilization procedure was not explained in detail. However, this procedure makes sense in this work in order to avoid the presence of other microorganisms different from that ones used in the flooding solution.

2.4.2 Group 2: Consolidation samples by compaction

Within the consolidated samples, compaction was identified as one of the methodologies with greater application. Here, a load to the plug can be exerted in a uniaxial or triaxial way. For example, by mixing particles of sand with shale in different proportions and applying a uniaxial compaction within a metallic cell. As a result, a consolidated rock sample can be obtained and used to analyse the dielectric constants of the rocks [31].

In this same way, it was proposed to obtain consolidated synthetic plugs by using unconsolidated reservoir rock samples. The samples were disintegrated manually [81] to guarantee the same particle size of natural unconsolidated rock. In the mentioned work, the aim was to compare the porosity, particle size and internal structure of unconsolidated and synthetic plugs. It was concluded that both samples had similar properties. In addition, it can be emphasized that in this way, it is possible to guarantee a natural sample reproduction.

2.4.3 Group 3: Consolidation by bonding materials

One of the first methodologies regarding the consolidation of plugs using bonding materials was reported in 1999 [35]. This consolidation was achieved by applying a uniaxial force into a mixture of quartz and epoxy resin, during the curing process of the epoxy. These constructed plugs were used to evaluate the compressive strength of the plugs. It is noteworthy that properties such as porosity and permeability of these plugs were not evaluated due to its geophysics application studies.

Subsequently, based on previous methodology [35], sandstone rock plugs were constructed by [71] aiming to simulate reservoir conditions and evaluate the xanthan gum effect on water production. Portland cement was used instead as a bonding material. The composition of the sandstone samples was 74% (w/w) sand, with particle sizes of 0.71 mm, 0.5 mm and 0.25 mm. The mixture consisted of 14.8% (w/w) Portland cement and 11.1% (w/w) water.

On the other hand, other authors developed an equipment called EDR – Radial Displacement Equipment [56, 63, 67], which allowed them to simulate the physical behaviour of water and steam injection processes in synthetic sandstone rocks. The equipment is composed by a square stainless-steel cell with an acrylic lid where the synthetic porous media was constructed using sand and polyester resin as a bonding material. For such, the materials were mixed and distributed homogeneously into the stainless-steel cell. The cell is closed during the resin setting time. After that, flooding tests were carried out using configuration composed by two wells: an injector well at one side and a producer well at the other side of the cell. The presented methodology allows the construction of sandstone rocks, however, without control of petrophysical properties.

Based on the previous methodology of [56], a methodology for plug construction with controlled petrophysics properties (porosity and permeability) was developed, by mixing different quantities of sand, white kaolin and resin epoxy in a cylindrical cell of polyvinyl chloride [63]. After the setting time of the kaolin and resin epoxy, consolidation and petrophysical properties of the samples were analysed by gas porosimetry. The authors concluded that a greater amount of binder material caused lower porosities and permeabilities. Additionally, porosities from 10% to 35% and absolute permeabilities from 13 000 mD to 0 mD and were related to the content of white kaolin. Reference [67] adapted the methodology used by [63] by using fixed quantities of sand, white kaolin and resin epoxy. The authors applied manual compaction procedure varying the number of hammer blows applied into a piston localized at a cylindrical cell of polyvinyl chloride. The control of petrophysical properties was not possible since the manually applied load by the operators was not controlled.

Therefore, using the same homogenized material of [67, 73] standardized the compaction process by applying controlled uniaxial torques, resulting in a greater reproducibility and repeatability of the petrophysical properties of the samples.

The authors of [75] focused their research on constructing electro-pneumatic equipment that produced a more controlled and higher compaction than those obtained by [73]. In this sense, the authors developed a methodology correlating the applied pressure and mixture composition (sand, white kaolin, epoxy resin and water). It was concluded that samples manufactured with 100 psi, using kaolin and sand with particle size of 0.149–0.074 mm, presented an excellent consolidation. Additionally, porosities between 21% and 33%, and permeabiities between 40 mD and 470 mD, were observed. Consequently, the authors recommended the construction of high pressure equipment to obtain samples with lower petrophysical properties.

Based on the mentioned works, it was observed that using resins for bonding materials generate well consolidated plugs that can be used in high pressure test such as gas porosimetry or fluid displacement tests. However, the presence of the resins could modify the behaviour of the sample in laboratory tests where surface chemistry has a key role, such as in wettability tests.

2.4.4 Group 4: Consolidation samples by material precipitation

Within the synthetic plug construction, the consolidation by precipitation of silica and carbonate materials was identified.

References [43, 32] describe precipitation, and subsequent consolidation, of silica by CO_2 flowing in a homogeneous sand and sodium silicate solution, which was subsequently subjected to uniaxial and confinement pressure. The authors concluded that samples had weak mechanical properties. In turn, they emphasize that due to the precipitated material quantity, it will be possible to control the petrophysical properties. This is because the increase of quantity of precipitated material reduces the porous space between the particles, decreasing the porosity and permeability.

From another consolidation perspective by calcium carbonate, two types of methodologies were identified: use of bacteria (biologically induced precipitation) and enzyme-induced precipitation. A biologically induced precipitation, with a petroleum engineering application, was described in [24]. This consolidation method employed a flow of solution consisting of Bacillus pasteurii NRS 673 bacteria, Difco nutrient, ammonium chloride, sodium bicarbonate, anhydrous calcium chloride and distilled water. Additionally, solvent and water stability precipitation were noted. The authors describe a permeability reduction on porous media, due to the carbonate precipitation. In addition, this paper emphasizes that this carbonate acted like two agents: cementer and obstructer.

Reference [88] described some of the improvements that are obtained implementing the biologically induced carbonate precipitation. In this sense, regarding construction of experimental plugs, the authors used sand with a density of 1560 kg/m^3 added into a suspension solution of ureolitic bacteria "sporosarcina pasteurii", urea and calcium chloride. After the treatment, five sample blocks were extracted by drilling, and cylindrical rock plugs with a fixed porosity value were obtained.

References [29, 30, 47, 57, 66] describe a controlled calcium carbonate precipitation process around silicon sand. This process simulated a natural cementation rock, and used a patented aqueous solution flow at low pressure. This method titled "CIPS – Calcite In-situ Precipitation System" allowed synthetic sample permeability control until a given condition.

Reference [37], based on the CIPS patent, developed a technique for artificially cemented calcareous soil preparation, looking for a high cementing quality and consistent density of the sample.

Besides, reference [66] creates another method, justifying the CIPS unknown patent solutions and the complex management of containment, extinction and generation of bacteria. The authors describe a precipitation method using urease, urea and calcium chloride. After testing this method, it was concluded that further studies are needed to understand how permeability and resistance can be restricted, controlling the amount of precipitated calcium carbonate.

Another method based on carbonate precipitation was described by reference [64], adapting the Brazilian standard "NBR 7182: Construction of compacted soil samples" [92] to construct a synthetic rock plug to study the behaviour of this sample in the presence of hydrochloric acid, aiming at carbon capture CO_2 storage studies. For this reason, a quartz sand sample was cemented using a homogeneous aqueous calcium hydroxide and calcified green algae (halimeda), using a proportion of 50%, 25% and 25%, respectively. Subsequently, the sample was compacted and carbonated with CO_2. From this work is important to note the author's emphasis on carbonate mineralogical reproduction, despite not reproducing petrophysical properties.

2.4.5 Group 5: Consolidation samples by materials sintering

Reference [93] describes a sintering process that involves a compacting heat treatment to transform powder into a solid material by the union of its particles. For this, heat treatments are required close to the material melting temperature. Some materials were found as a basis of consolidation by thermal sintering of materials: Polytetrafluoroethylene (PTFE) or Teflon and quartz.

Reference [16] describes the methodology for constructing plugs by using PTFE or Teflon particles previously compressed in a mould, where a temperature of 371 °C was applied for a sintering process to occur. Reference [20] explains that Teflon guarantees an inert chemical composition and low surface energy, resulting on wettability control.

References [16, 94, 95] indicate that porous media and surface of sintering PTFE samples appear like sandstone rocks. They claim this due to its pore structures at microscopic scale, rough and irregular surfaces, and uniform porosities and permeabilities. However, they did not elaborate on the surface chemical composition.

Finally, reference [25] describes the production of porous models by quartz particle sintering, with particle sizes between 0.04 mm and 0.07 mm. The authors justify the use of this technique due to the easy cleaning procedure

and reuse advantage. No concerns about mineralogical and petrophysical parameters control were considered.

In a summary, the construction of synthetic plugs is viable and dependent on the selected materials and methodologies. Therefore, there is a lack of work focusing both mineralogical composition and petrophysical properties. In this way, this work also presents a promising approach to obtain such plugs, as will be discussed in the following section.

3 Proposal for carbonate plug construction

Plug composition has a direct relationship with the rock chemistry, and therefore with the behaviour of the sample in the laboratory tests focused on the surface chemistry behaviour, such as wettability measurements aimed at enhanced oil recovery studies. According to reference [96], wettability refers to the preference of a solid surface to be in contact with a fluid, due to the equilibrium of interstitial forces (fluid-solid). In the case of a petroleum reservoir, these forces will generate a system with preferences to be oil-wet or water-wet due to the behaviour between oil, water and reservoir rock. This means that due to the wettability, if reservoir rock is oil-wet, higher amounts of oil can remain retained inside the reservoir rock, resulting in a negative economic situation [4]. For example, in the case of sandstone rocks, preferences to be water-wet [97, 98] were found, and carbonate rocks to be oil-wet [6, 99, 100] or with an intermediate wet behaviour [101, 102] were found.

Because these plugs can be also used for advanced petrophysics studies, such as flooding or wetting measurements, it is important to choose materials which allow synthetic carbonate samples to be built, guaranteeing their similarity to the natural ones, such as in studies of changing wettabilities due to the application of chemical treatments to the rock-fluid system in order to enhance oil production.

To achieve carbonate reservoir samples that reproduce the proper mineral chemistry composition, materials based on silica's and sand, as well as bonding materials based on epoxy, resins and cements, will not be considered. Therefore, based on the developed methodologies by [42, 81], which used poorly consolidated rock samples that were manually disintegrated and used as base material to obtain synthetic core plugs, an adaptation is proposed that uses a disintegrated dolomitic carbonate rock. In this way, disintegrated carbonate rock will be the unique conformant material of synthetic plugs, guaranteeing a similar natural composition compared to the corresponding reservoir rock. However, in this work a consolidated carbonate rock was used, and the disintegration procedure was done by using equipment which allowed to control the particle sizing of the base material.

Table 3 presents the chemical composition of the used dolomitic carbonate, obtained by X-ray spectrometry fluorescence in the STD-1 (Standardless) calibration, concerning the analysis of chemical elements between fluorine and uranium. The Loss on Ignition (LOI) was carried out at 1020 °C for 2 h. It is emphasized that according to reference [103], the high concentration of the compounds MgO (15.5–17.2%), SiO_2 (4.51–4.06%) and CaO (36.4–33.8%) corresponds to dolomitic calcareous samples. In this way, dolomitic carbonate rock samples were disintegrated and classified using the following steps. Disintegration of the natural sample was carried out by using a jaw crusher and a roller crusher, until the desired particle size was obtained, which was controlled by a sieve. Based on the particle size analysis (Fig. 3), a particle size control in five intervals around the most used granulometry was proposed, aiming to obtain plugs with different basic petrophysical parameters. They were: 0.3 + 0.6 mm, 0.15–0.3 mm, 0.074–0.15 mm, 0.020–0.074 mm and <0.020 mm.

Table 3. Chemical composition of the dolomitic carbonate used to construct synthetic plugs.

Chemical composition	Sample 1	Sample 2
Na_2O	<0.001	0.059
MgO	15.5	17.2
Al_2O_3	0.765	0.652
SiO_2	4.51	4.06
P_2O_5	0.014	0.018
SO_3	0.033	0.089
Cl	0.021	0.014
K_2O	0.064	0.205
CaO	36.4	33.8
TiO_2	0.043	0.028
Cr_2O_3	0.011	0.012
MnO	0.103	0.046
Fe_2O_3	0.564	0.579
NiO	<0.001	0.006
ZnO	0.024	<0.001
SrO	0.018	0.014
ZrO_2	0.044	0.002
Nb_2O_5	0.029	0.014
LOI	41.9	43.2

In addition, it was necessary to pulverize quantities of 0.02–0.074 mm granulometry to obtain the finest particle (<0.020 mm) using a Herzog pulveriser. To corroborate the true size of the material resulting from this procedure, a low angle laser light scattering measurement (Malvern Mastersizer) was used to assure particle size. As a result, two graphs were obtained (Fig. 5). The first one was the percentage of volume *versus* size of particle, and the second one was the accumulated percentage of the volume analysed. From this figure, it can be determined that according to the distribution of particle size, the material analysed has a particle size less than 0.020 mm. Therefore, it was concluded that the described procedure of pulverization of the sample is correct to obtain this specific finest particle. The particles were not submitted to a drying procedure.

Next, a uniaxial compression method was used, at first, to verify the resultant consolidation of the selected particle sizes. For this reason, a 200 kN load force was applied two times, by using a mechanical hydraulic press on 5 g of material. The dimension of the resultant disc was 38 mm in diameter and 2 mm in length. Figure 6 presents this

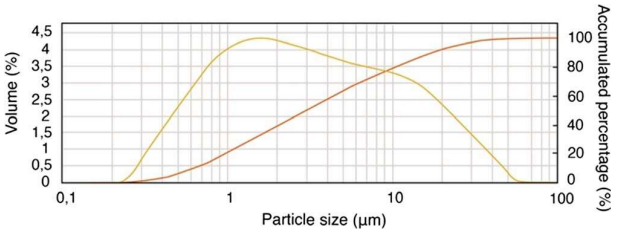

Fig. 5. Particle size distribution of pulverized fraction.

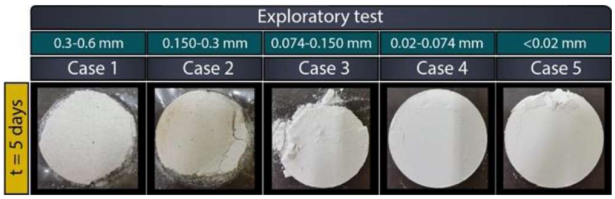

Fig. 6. Exploratory test discs aiming to study the influence of particle size on material consolidation and drying temperature 25 °C.

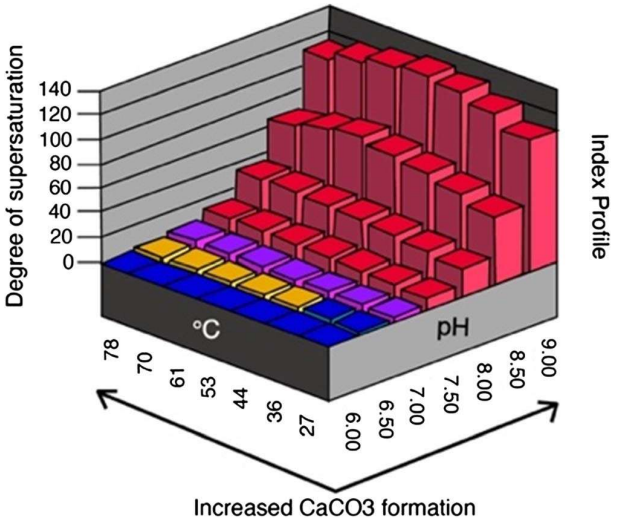

Fig. 7. Degree of supersaturation – $CaCO_3$ formation. Extracted from reference [104].

exploratory samples after 5 days of their production. From this test, fragile discs with varying consolidation were observed, which was improved with a decrease in the particle sizes. This consolidation behaviour can be explained due to favourable material agglomeration. This first exploratory test demonstrated a promising preliminary result regarding the construction of real size plugs.

Based on the previous results, it was necessary to find an additional methodology to guarantee samples with better consolidation. Thus, based on the known increase in calcium carbonate solubility being related to both pH and decreasing temperature (Fig. 7), it was decided to apply temperature and controlled pH water drops on the construction process of synthetic plugs, as follows.

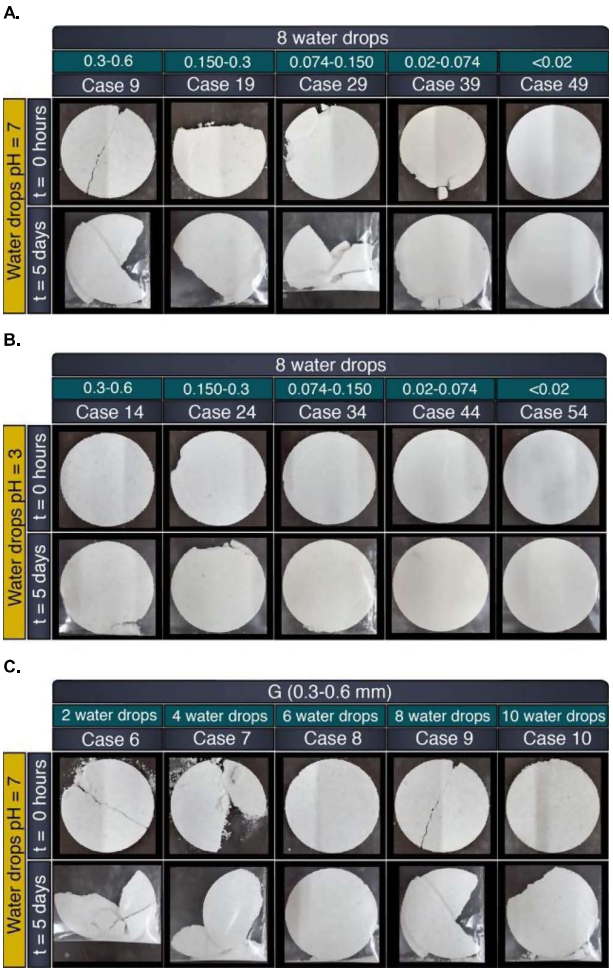

Fig. 8. (A) and (B) Resulting discs from decreasing particle size, 8 water drops, drying temperature 25 °C and pH values 7 and 3 respectively. (C) Resulting discs from particle size 0.3–0.6 mm, 2–10 water drops of pH 7 and drying temperature 25 °C.

Aiming to evaluate the effect of these new parameters on plug consolidation, the pH effect was verified by adding distilled water drops with pH values of 3 and 7 (from 2 to 10 drops) to the 5 g particle size material, followed by mixing for 1 m before the compression process. The acidity of the water drops was achieved by using diluted hydrochloric acid solution.

The mixture was submitted to the same uniaxial compression used on previous exploratory tests. Load force and compression times used were fixed aiming to guarantee an experimental reproducibility, and compare the resulting plugs with exploratory ones. These samples were dried at room temperature (25 °C) with a range of humidity between 60% and 80%. Figures A.1–A.5 in the annexe present all of the constructed discs, at 0 h and 5 days after the consolidation process. Figure 8 presents some of the resulting discs, in order to illustrate the following analysis.

The consolidation behaviour, again, present a tendency to improve with decreasing particle size. Figures 8A and 8B

present this improved consolidation corresponding to the decrease of particle size for discs with the same number of water drops with a pH value 7 and 3. In addition, it was noted an effect of the time of sample drying on the consolidation behaviour, getting worse over time. It is due to temporary sample consolidations caused by the presence of humidity. Figures 8A–8C present this consolidation behaviour. On the other hand, comparing the Figures 8A and 8B, a possible calcium carbonate dissolution when using acid water drops (pH 3), followed by its probable precipitation, led to a more effective consolidation.

Comparing cases with the same granulometry, drying times and number of water drops with pH 3 and 7 from Figures A.1–A.5 in the annexe, it can be seen that a greater number of samples remained consolidated, even after 5 days. In this way, the use of acid water drops would be the most beneficial way for the construction of samples, even when using larger particle sizes.

Regarding the number of water drops, it was observed that few (2 and 4 drops) or more (8 and 10 drops) did not generate good material consolidation (Fig. 8C). This trend 'may be due to the amount of water not being enough to impregnate all of the particle surface, or the quantity of water being too much to allow good material handling, turning it into a very weak sample. Therefore, with 6 water drops the material was not excessively humid, and the total impregnation of its particles was possible, causing a homogeneous solubility (in the case of acidic pH) and material consolidation. Finally, 6 water drops, pH = 3 and particle size <0.02 mm seemed to generate the best disc consolidations.

The constructions of the disks were performed by using a drying temperature of 100 °C for 1 h and the controlled pH water drops, as explained previously. Figures A.6–A.10 in the annexe present these constructed discs at 0 h (after removing from compression moulding), 1 h (after drying process at 100 °C) and 5 days (after the consolidation process). From these samples, it was observed a positive influence on the consolidation of the material due to high drying temperatures, comparing discs with the same particle size, pH and number of drops. In this sense, samples with high drying temperatures (100 °C) present a tendency to have a better structure than those with dried at room temperature (25 °C). Figure 9 exemplifies this behaviour, from a comparison of constructed discs at 5 days, with particle size (0.074–0.150 mm), 2–10 water drops, pH value 7 and drying temperatures of 25 °C and 100 °C for 1 h. In addition, it is believed that due to the high temperature (100 °C), a smaller amount of water was retained within the sample. This made it possible to guarantee a drier and more definitive consolidation.

Finally, given the previous findings, plugs of a real size (25.4–38.1 mm in diameter and 1–1.5 times greater than the diameter in length) were produced. For this reason, the construction conditions selected to guarantee a good consolidation were: the finest particle size material (<0.020 mm), six water drops at pH 3 and drying temperature of 100 °C. Based on 2 mm length discs obtained from an applied load of 200 kN on 5 g of material, 100 g was the required material quantity to obtain plug lengths of about 40 mm at the same applied load. In addition, 120 water drops were added to the material and the compressed samples were subjected to the 100 °C drying temperature for 1 h. The first synthetic plug of real size is presented in Figure 10. The dimensions of this plug were 37.7 mm in diameter and 39.5 mm in length.

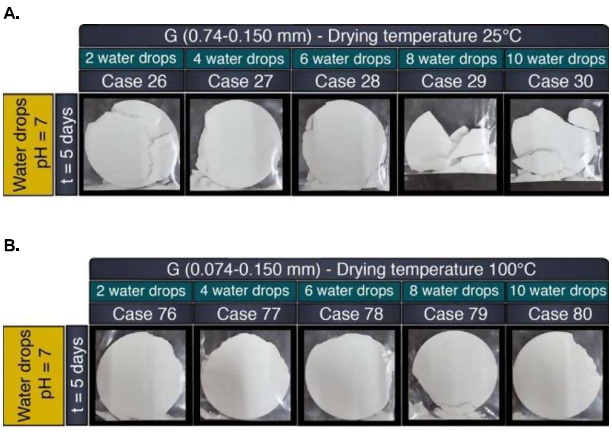

Fig. 9. Resulting discs at 5 days, constructed with particle size (0.074–0.150 mm), 2–10 water drops, pH value 7. Drying temperatures (A) 25 °C and (B) 100 °C for 1 h.

Fig. 10. Synthetic carbonate plug of real size, using particle size (<0.020 mm), six water drops with pH 3, uniaxial compression and drying temperature 100 °C for 1 h.

In the synthetic sample, the presence of wax due to the lubricating material used on the compression piston can be observed. On the other hand, scratches on the lateral surface were observed, which seem to compromise the structure of the material. These samples were sanded (sandpaper No 600), where it was observed that the scratches were superficial and therefore, did not affect the internal structure of the sample. It is notable that this sample presented a good consolidation before and after the sanding, confirming a good choice of the construction materials and method.

Aiming to evaluate the basic petrophysical properties, samples were submitted to porosity and permeability gas (nitrogen) test with a confinement pressure of 2400 psi in an Ultrapore 300 porosimeter and Ultraperm 500 permeameter, both from Corelab. For the synthetic plug, a porosity of 25.19% $\pm$ 1% was obtained, and mean absolute permeability was 0.073 mD $\pm$ 0.022 mD. This high porosity and low permeability can be explained as the result of small internal pores in the structure of the sample. On the other hand, due to the undamaged condition of the samples after the application of the high confinement pressure of 2400 psi, it was concluded that sample presented good consolidation. Additionally, grain density of this synthetic plug was calculated in order to compare the dolomite rocks value of 2.84 g/cm^3 from literature [105]. In this sense, it was found a grain density for this synthetic plug of 2.87 g/cm^3, corroborating the similarity of the synthetic sample with the natural ones.

It is important to mention that this work is a preliminary part of research project, where material correlations and construction methodologies have been studied. The main contribution to the area is the identification and adaptation of the methodologies developed up to now, by means of a plug construction proposal that correlates the study of the materials and methodologies, for the generation of knowledge regarding the behaviour of carbonate reservoir rocks.

The difficulty of obtaining reservoir plug samples and the economic importance they possess are well known. This study proposes an alternative for the construction of synthetic plugs that can be used as study samples for the evaluation of physical and chemical tests in the laboratory, targeting increases related to oil production.

The correlation between particle size and petrophysical properties, such as porosities and permeabilities, is expected to be further studied by the combination of controlled quantities of the different granulometries. In addition to this, future studies will be based on the use of drilling reservoir rock cuttings as the main conformant material of the synthetic samples, in an effort to reproduce samples closer to the natural ones. On the other hand, studies between the uniaxial pressure ratio and petrophysical properties of the rock are necessary, as an additional parameter to control during the construction of future synthetic samples.

Further tests are needed to develop procedures where the petrophysical properties can be more controlled, in addition to the mineral composition. Effects of particle size distribution and the pressure applied on the porosity and permeability of the obtained plugs are some examples. Finally, to avoid scratches on lateral plug surface, controlled and lower compression velocities should be applied.

4 Conclusion

The paper presented an extensive review of the literature, compiling the most notable studies on the construction of synthetic plugs. Specifically, after looking for the reproduction of carbonate samples, a lack was found on methodologies that approach this type of rock. Some of the work aimed to study the consolidation process using precipitation methodologies, which would be more appropriate to simulate the mineralogical matrices closest to such reservoirs. In this sense, the use of resins or cement as binder materials would not be the most suitable for the cases of special petrophysical studies.

An adaptation of existing methodologies was proposed by means of the implementation of an uniaxial compaction of pulverized carbonate type rock with different particle sizes, changes in temperature and pH. About the construction of the preliminary samples, the best consolidation seems to be related to the followings conditions: by decreasing the grain size of the matrix elements (particle size <0.02 mm), number of pH 3 water drops (6 drops/5 g of material) and drying temperature (100 °C). The samples obtained showed promising results for the reproduction of plugs of real size and similar mineralogy to the natural carbonate reservoir samples. However, further studies are necessary to obtain a more controlled petrophysical properties.

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Annexe

Fig. A.1. Constructed discs aiming to study the influence of pH and quantity of water drops on the carbonate consolidation. Particle size (0.3–0.6 mm) and drying temperature 25 °C.

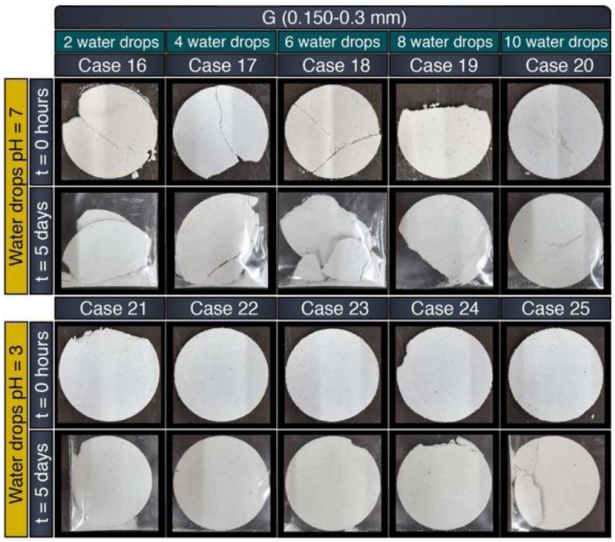

Fig. A.2. Constructed discs aiming to study the influence of pH and quantity of water drops on the carbonate consolidation. Particle size (0.150–0.3 mm) and drying temperature 25 °C.

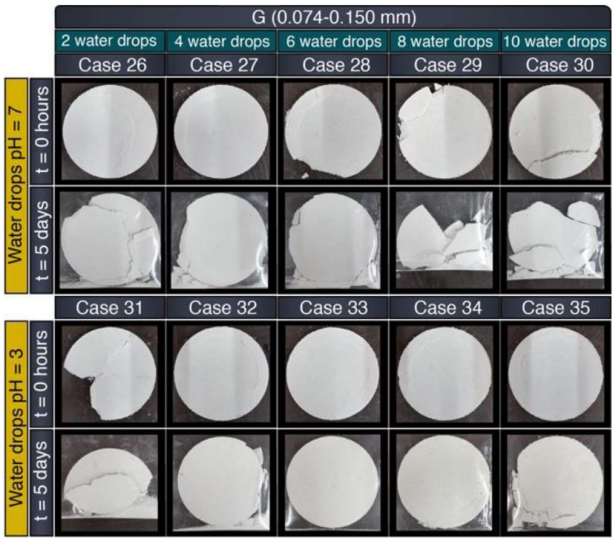

Fig. A.3. Constructed discs aiming to study the influence of pH and quantity of water drops on the carbonate consolidation. Particle size (0.074–0.150 mm) and drying temperature 25 °C.

Fig. A.4. Constructed discs aiming to study the influence of pH and quantity of water drops on the carbonate consolidation. Particle size (0.020–0.074 mm) and drying temperature 25 °C.

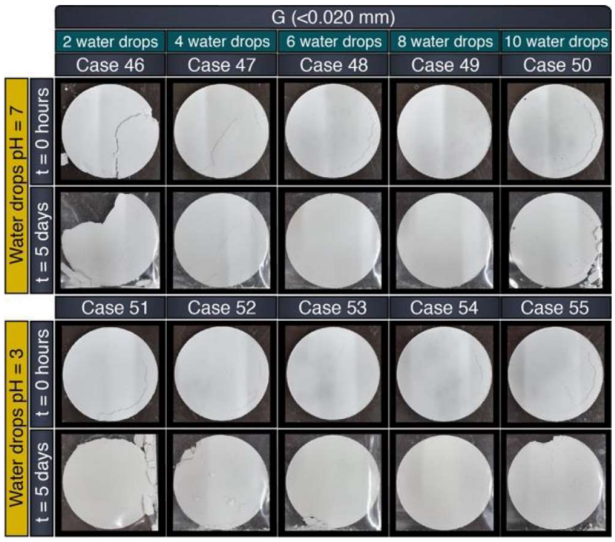

Fig. A.5. Constructed discs aiming to study the influence of pH and quantity of water drops on the carbonate consolidation. Particle size (<0.020 mm) and drying temperature 25 °C.

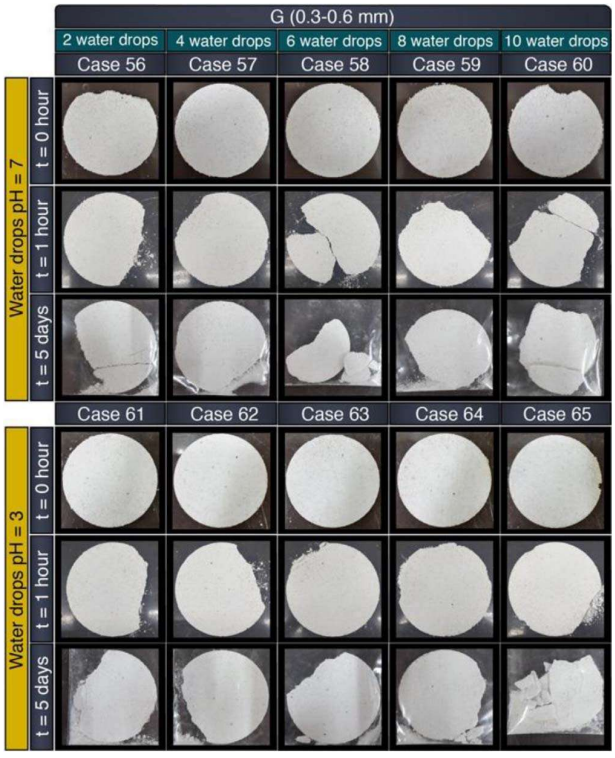

Fig. A.6. Constructed discs aiming to study the influence of temperature on the carbonate consolidation. Particle size (0.3–0.6 mm) and drying temperature 100 °C for 1 h.

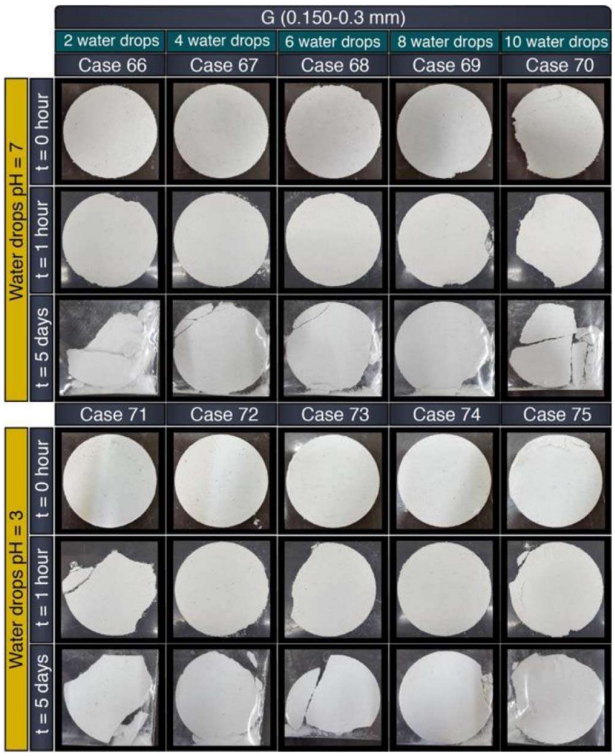

Fig. A.7. Constructed discs aiming to study the influence of temperature on the carbonate consolidation. Particle size (0.150–0.3 mm) and drying temperature 100 °C for 1 h.

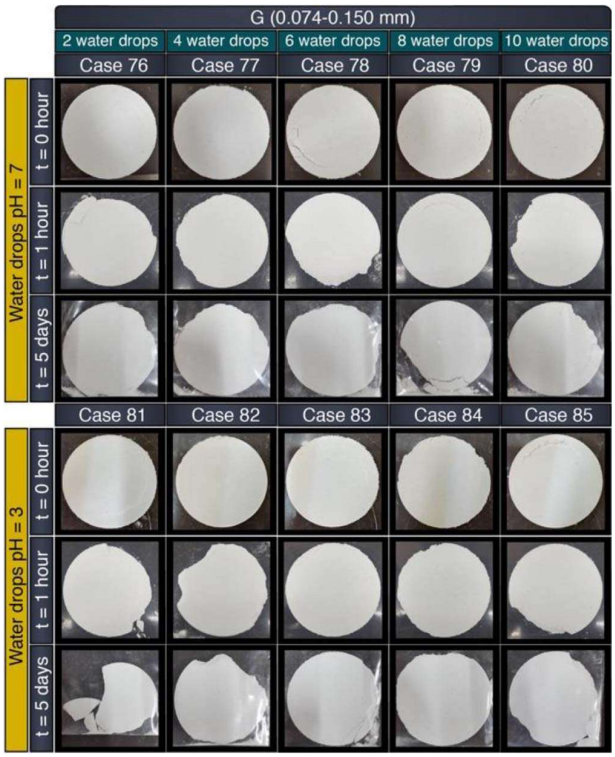

Fig. A.8. Constructed discs aiming to study the influence of temperature on the carbonate consolidation. Particle size (0.074–0.150 mm) and drying temperature 100 °C for 1 h.

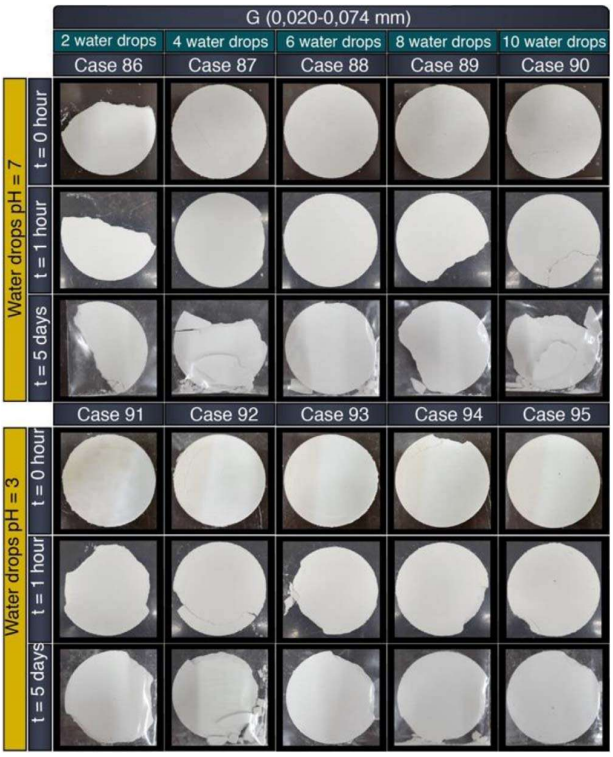

Fig. A.9. Constructed discs aiming to study the influence of temperature on the carbonate consolidation. Particle size (0.020–0.074 mm) and drying temperature 100 °C for 1 h.

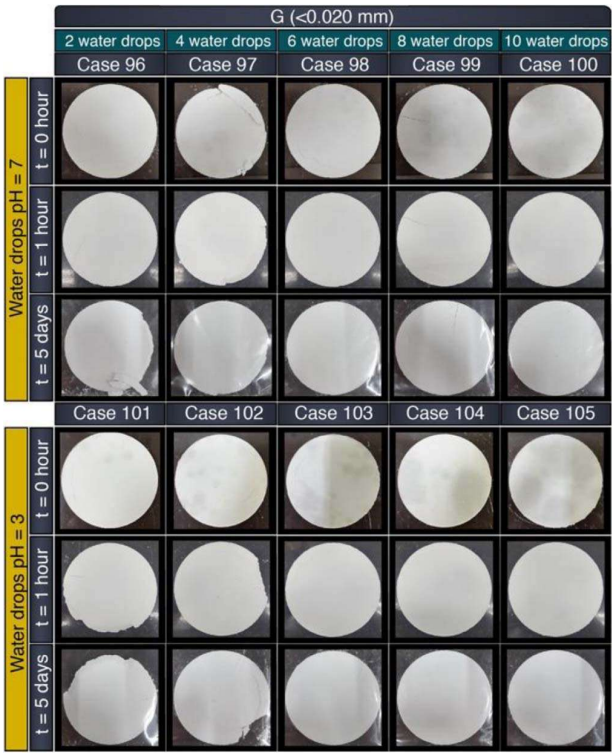

Fig. A.10. Constructed discs aiming to study the influence of temperature on the carbonate consolidation. Particle size (<0.020 mm) and drying temperature 100 °C for 1 h.