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USE OF MICROCAPSULES TO CONTROL GELATION OF POLYMERS USED TO BLOCK FRACTURES IN
OIL RESERVOIRS

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Abstract. The efficiency of oil displacement is greatly reduced by the presence of fractures and high permeability layers. The injected water phase flows preferentially through low resistance paths, leaving large volumes of the reservoir not affected by the water injection process. An alternative to minimize this problem is to block fractures with a liquid system that forms a gel after some time, forcing the water phase to flow through the porous matrix. Sodium silicate gel is one of the formulations that can be used for this purpose. However, the kinetics of the silicate gel formation process is difficult to control, since it depends on many different variables. This uncertainty of the gelation process may lead to the formation of a gel phase away from the desired position, leading to many different problems, such as loss of injectivity. A liquid formulation using microcapsules is designed to control the release time of the activating agent and, consequently, to improve control over the start of the gelation process. The first step in the development of this technique was to study evolution of the rheology of the gel formed from sodium silicate with hydrochloric acid in order to evaluate the rate of the gel formation as a function of different process parameters, such as the concentration of Na-Si and HCl. The results show that the gelation process is a strong function of the solution pH and that the gelation time is shorter the higher the concentration of hydrochloric acid. In the proposed method, the hydrochloric acid solution is encapsulated and the gelation only starts after the bursting the capsule shell, which is triggered by imposing a gradient of osmotic pressure.

Keywords: fractured reservoir, gel systems, sodium silicate, rheology, microcapsules, microfluidics

1. INTRODUCTION

Oil recovery involves a series of costly operations aimed at extracting as much oil as possible from the reservoir. Historically, oil recovery methods have been basically subdivided into three stages: primary, secondary and tertiary, conveying a chronological idea of intervention in reservoir production (Musse & Quintella, 2009).

The terms “primary” and “secondary” refer to the beginning of the oil industry, when reservoirs were initially produced without injecting fluids, until reaching the minimum economic flow rates in producing wells; this production phase was known as “primary” (Ferreira, 2016). This consists of oil extraction by natural mechanisms, which depend on reservoir conditions such as volume, porosity, permeability, pressure and temperature (Lima, 2015). In the second phase, equipment and wells associated with the injection of gas and/or water were installed to supply additional energy to the reservoir, with the main objective of maintaining the pressure and displacement of the oil by the injected fluid. Therefore, this technology was associated with a “secondary” phase, illustrated in Figure 1. The term tertiary recovery is associated with processes performed after an injection of water or gas. However, these technologies can also be used at the beginning of field production, as usually occurs in cases with the application of thermal methods. Therefore, the term “tertiary recovery” is not suitable for many cases, and in more recent publications it has been replaced by “improved recovery” or “advanced recovery” (Ferreira, 2016).

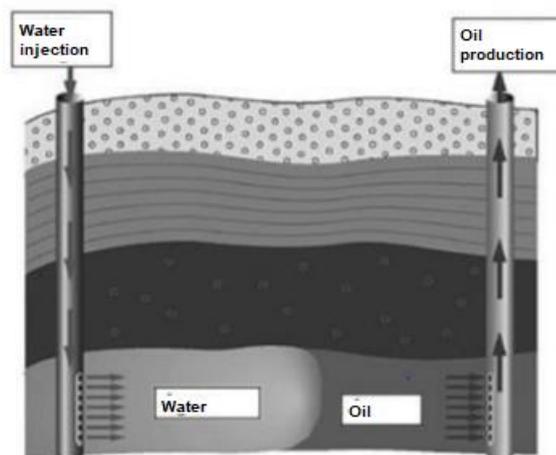


Figure 1: Injection of water into an oil reservoir. Source: (Cajazeiras, 2018)

The use of water injection process is responsible for more than half of the world's oil production, but this methodology has a limited reservoir sweeping efficiency, normally leaving a considerable amount of oil in the reservoir. This occurs, among other reasons, due to the unfavorable mobility ratio between water and oil, caused by the lower viscosity of water compared to oil, in most reservoirs (Lima, 2015). The Mobility Ratio, M , is given by:

$$M = \frac{(k_{water}/\mu_{water})}{(k_{oil}/\mu_{oil})} \quad (1)$$

Where:

k_{water} is the effective water permeability;

k_{oil} is the effective permeability to oil;

μ_{water} is the viscosity of water;

μ_{oil} is the viscosity of the oil (Lima, 2015).

The high mobility ratio M leads to the appearance of a phenomenon known as viscous fingering, which is the formation of patterns in a morphologically unstable interface between two fluids in a porous medium. The displacement front becomes unstable, forming preferential paths for the aqueous phase, as shown in Figure 2.

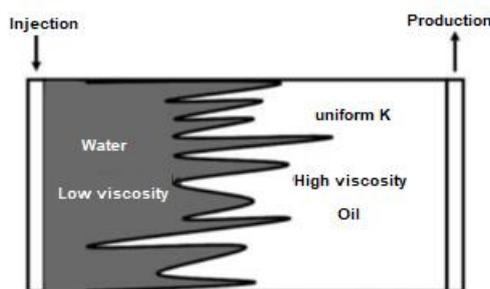


Figure 2: Viscous fingering phenomenon. Source: (Santos, 2010)

Overproduction of water, especially in mature oil fields, has serious implications for field operations and the environment into which produced water can be disposed of. Produced water is separated at the bottom of the well and injected into another formation or brought along with oil/gas to the surface and separated there. Operating expenses, including lifting, separating, treating, pumping and reinjecting and/or disposing of it, increase the overall cost of oil production. Once water is separated from oil, it can be reinjected into the formation or discharged into the environment. In 2002, it was estimated that the water produced would cost the oil industry approximately US\$45 billion annually (Pham & Hatzignatiou, 2015).

In the case of fractured reservoirs, the problem of non-uniform oil displacement is even more critical, as the injected water preferentially flows through the fractures. If the fractures are close to the injection wells, almost all the injected water flows through the fractures, strongly compromising the efficiency of the entire oil displacement process. In a more general definition, a fracture is any discontinuity within a rock mass that developed in response to stress (Bonnet et. al., 1999). The presence of fractures has a strong effect on the behavior of naturally fractured reservoirs, as they present

abrupt changes in the porosity and permeability properties of the medium, acting as walls, or preferential paths for the flowing fluids (Genesis, 2018). In the case of fractured reservoirs, the problem of non-uniform oil displacement is even more critical, as the injected water preferentially flows through the fractures. If the fractures are close to the injection wells, almost all the injected water flows through the fractures, strongly compromising the efficiency of the entire oil displacement process.

1.2 Motivation

Some solutions can be applied to delay production and control the excess volume of water brought to the surface due to preferential flow through fractures close to the injection wells. Reducing water production greatly reduces the total costs of fluid production (surveying, produced water treatment, storage and reinjection), the environmental impact of oil and gas operations, and risks associated with reinjection wells. Injection of gelation polymers can be used to increase the viscosity of the injected water, blocking the preferential pathways and preventing the early breakthrough of the water, improving the formation's sweeping efficiency (Pham & Hatzignatiou, 2015). According to Portwood (Portwood, 1999), a typical water block treatment could reduce water production by 75% to 90%.

Chemical solutions have a wide range of application in terms of depth and can solve many water-related problems. Appropriate chemical systems can be used either in the area close to the well to block the most productive layers of water (with greater efficiency compared to mechanical techniques) or in-depth treatments to block fractures originating far from a production well (Pham & Hatzignatiou, 2015).

Polymer gels and sodium silicate are the most popular chemicals for blocking water. These products are injected as solutions to form gels in the reservoir (Pham & Hatzignatiou, 2015). Gels are designed to be strong enough for long periods at the temperature, salinity and pH of the formation; in addition, they are able to withstand the pressure applied during the production of hydrocarbons. The resulting profile modification or compliance control diverts injected water to unswept reservoir zones and improves fluid distribution in heterogeneous reservoirs (Simjoo et. al., 2007). As a result, a greater volume of recovered oil can be obtained. An illustration of the mechanism is shown in Figure 3. Before gel insertion, the recovery fluid follows preferential paths, such as fractures, leaving pores unswept. With the gel, you can block these paths and force the fluid to travel through spaces that have not yet been swept.

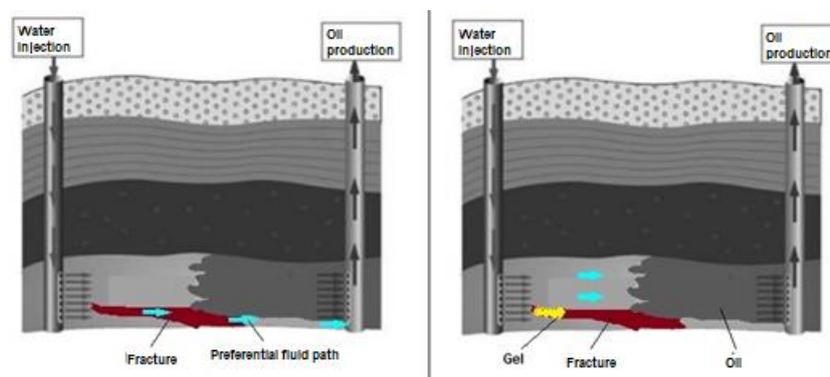


Figure 3: Illustration of Fracture Lock Mechanism to Change Preferred Recovery Fluid Path. Adapted from: (Cajazeiras, 2018)

However, the kinetics of the silicate gel formation process is difficult to control as it depends on many different variables such as temperature and pH of the medium. This uncertainty in the gelation process can lead to the formation of a gel phase out of the desired position, leading to several problems, such as loss of injectivity. To avoid obstacles, it is important to develop a system that allows greater control of the gelation reaction, making it possible to deeply block fractures.

In addition, an important feature to be studied is the ease of removing the gel. A poor positioning inside the reservoir and not removing it could cause loss of injectivity or even obstruct the path of the drilling fluid in the incorrect location and cause serious problems such as kick and blowout. The flow of fluids, gas, oil or water, from the rock into the well, also known as a kick, occurs when the pressure exerted by the drilling fluid is lower than the formation pressure (pre-existing pressure in the pores of the rock to be perforated). When there is a failure to control this problem, a blowout can occur, that is, an uncontrolled flow of fluid from the formation to the surface (Costa & Lopez, 2011), which can even result in the destruction of the well, a financial loss of enormous proportions. One of the best-known blowout accidents is that of the Deep Water Horizon Platform, in the Gulf of Mexico, on April 20, 2010. The blowout was followed by explosions and fire, causing the death of eleven people and seventeen injuries. The platform sank two days later. The drilling well was out of control for three months and nearly five million barrels of oil leaked, causing a massive environmental disaster (de Castro, Martins, & Ferreira, 2015).

The objective of this work is to propose a process that allows a greater control of the beginning of the gelation of a sodium silicate system through the use of microcapsules. The basis of the process is the encapsulation of the reaction activating agent and its delivery, through the disruption of the microcapsule membrane, only after the system is in the desired location. The process would thus avoid the problems associated with gelation out of the desired position and consequent loss of injectivity as well as the gelation process of deep fractures.

The behavior of the Na-Si gel combined with the 2M hydrochloric acid activator was studied in order to broaden the knowledge about this mechanism. The concentrations of both components were varied, the viscosity profiles over time were mapped, as well as the elastic and viscous modulus curves.

Finally, the activating agent (hydrochloric acid) was encapsulated for gelation tests in contact with sodium silicate, demonstrating that the reaction only occurs after the rupture of the capsule shell, which is triggered by the imposition of an osmotic pressure gradient.

In chapter 2 of this work, the study of gel formation kinetics is presented, by the results of the experiments. In chapter 3, the tests' results of the use of microcapsules to control de gelation of sodium silicate are shown. Finally, the conclusions of this work are described in chapter 4.

2. STUDY OF GEL FORMATION KINETICS

In all experiments, the Sigma-Aldrich sodium silicate solution was used, whose composition is 10.6% Na_2O and 26,5% SiO_2 totalizing 37.1% of sodium silicate; and the density is 1.39 g/ml at 25°C. The activator used was hydrochloric acid with a 2M concentration. For dilution, deionized water.

In the viscosity tests as a function of time and in the oscillatory tests (measurement of elastic and viscous modules), a TA Instruments model DHR-3 rheometer was used. These tests were executed to characterize the evolution of the gelation process.

The tests were performed with samples containing 1g, 2g, 3g and 4g of sodium silicate solution, with concentrations of 3.3, 5.3, 7.8 and 9.0 wt% respectively, where 2M HCl was added gradually for control of the pH. Sigma-Aldrich's Na-Si solution already contains 62.9wt% water, but more deionized water was added to standardize concentrations. The total amounts and pH measurements before the addition of HCl are presented in Table 1 and all tests were performed at 25°C. In this table, the water concentration corresponds to the total from the Sigma-Aldrich commercial solution added to the addition of deionized water made in the tests.

Table 1: pH measurements of samples before 2M HCl addition

Na-Si [wt%]	Water [wt%]	pH
3.3	96.7	11.71
5.3	94.7	11.7
7.8	92.2	11.92
9.0	91.0	11.4

In this chapter, the experimental results will be presented. Different types of characterization tests were performed to understand the most suitable for proof of gel formation.

2.1 Viscosity tests with HCl variation

In order to analyze the effect of the amount of hydrochloric acid on the gelation of sodium silicate solutions, its percentage by mass was increased while the amount of deionized water was decreased to keep the Na-Si concentration constant. Table 2 shows the concentration of Na-Si, 2M solution of HCl and water systems used to form the gels.

Table 2: Activator amount for each sodium silicate sample

Na-Si [wt%]	HCl 2M [wt%]	Water [wt%]
3	10	87
	11	86
	11.2	85.8
	11.5	85.5
	12	85
4.6	12.5	82.9
	13	82.4
	13.3	82.1
	13.6	81.8
	14	81.4
	14.5	80.9
6.7	15	78.3
	15.7	77.6
	16.4	76.9
	17.2	76.1
7.7	15	77.3
	15.8	76.5
	16.6	75.7
	17.4	74.9
	18	74.3

The results of the viscosity tests as a function of time at a constant shear rate of $10s^{-1}$ with variation of 2M HCl's amount are presented in Figure 4.

As the system starts to form the gel, the viscosity increases, reaching values corresponding to 100x the initial viscosity. We arbitrarily consider the gelation time to be the injection point of the viscosity curves. In general, the gelation time falls as the concentration of HCl increases.

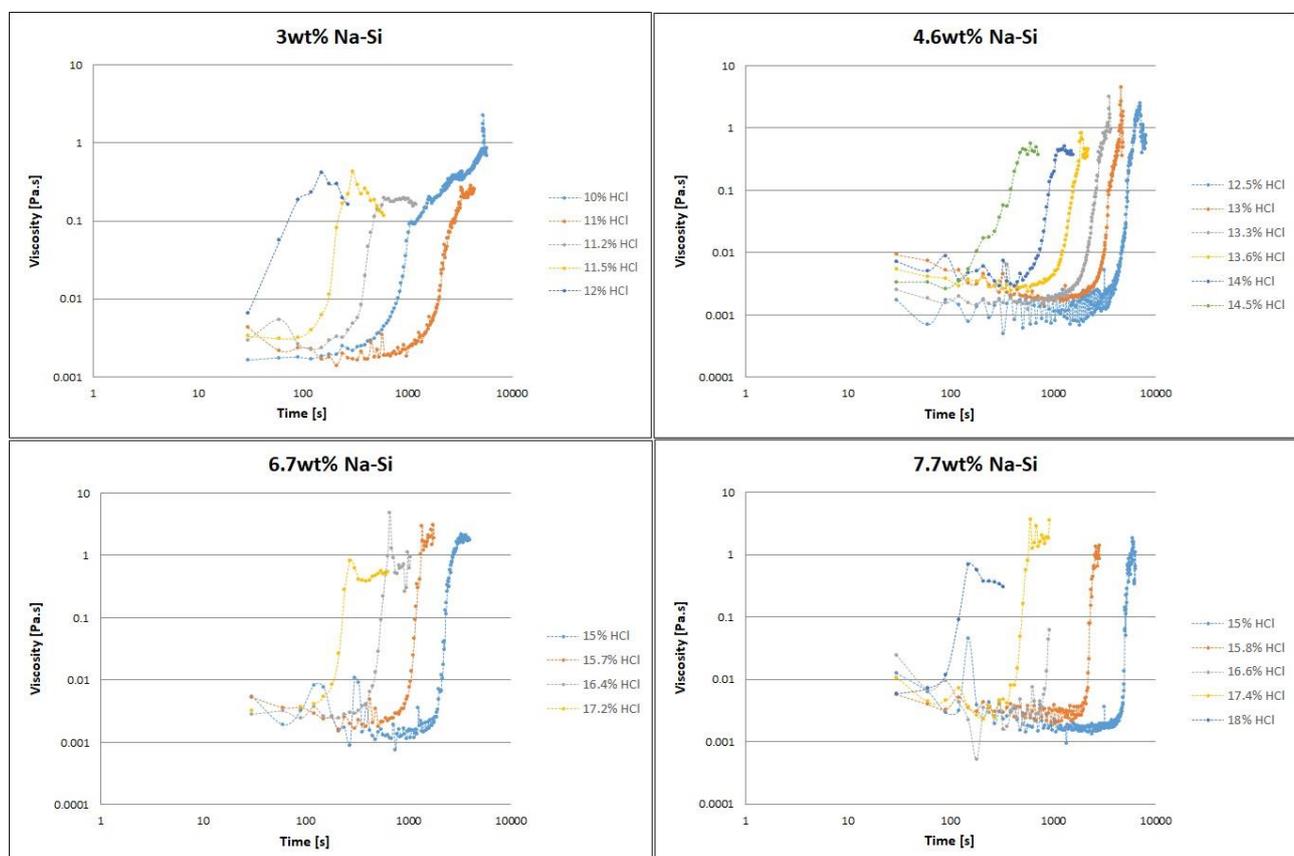


Figure 4: Viscosity versus time for each Na-Si sample with 2M HCl variation

Figure 5 shows the gelation time as a function of sample pH for each concentration of sodium silicate tested. It can be observed that pH is an important trigger in the formation of Na-Si gel. The lower the pH, the shorter the gelation time.

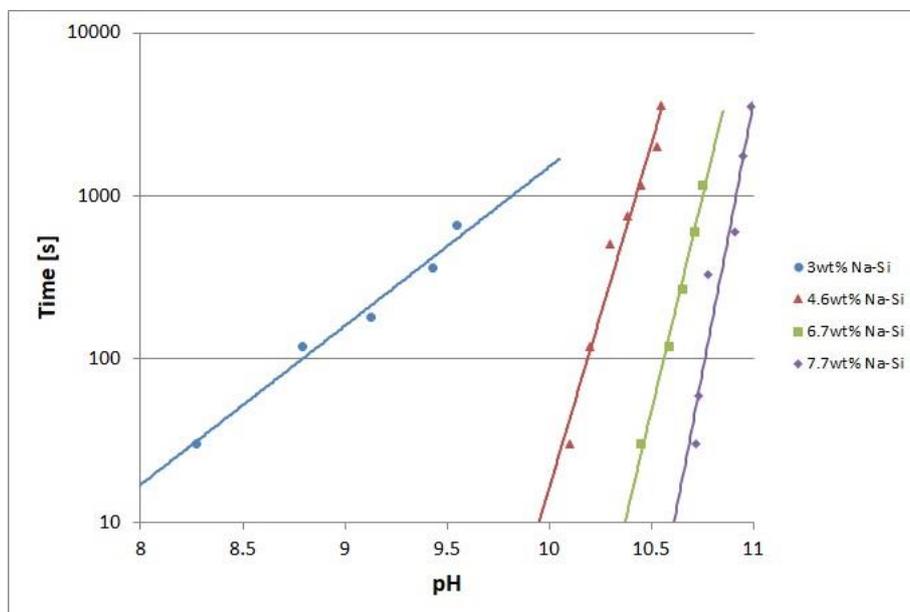


Figure 5: Gelation time of Na-Si as a function of pH

The tests for viscosity as a function of time are simple. Therefore, it was decided to perform oscillatory tests, which are more accurate, and the definition of gel formation time is taken as the instant at which the elastic modulus surpasses the viscous modulus in an oscillatory shear test.

2.2 Oscillatory tests

In these experiments, the elastic (G') and viscous (G'') modulus of the sodium silicate samples were measured with the amounts of 2M hydrochloric acid activator shown in Table 3. It was decided to carry out these experiments with the minimum concentration of HCl because the gelation process takes place more slowly, making it possible to capture the transformation more completely.

Table 3: pH measurements of samples after 2M HCl addition

Na-Si [wt%]	HCl 2M [wt%]	Water [wt%]	pH
3.0	10.4	86.6	9.43
4.6	12.5	82.9	10.45
6.7	14.9	78.4	10.65
7.7	15.0	77.3	10.76

First, strain scanning was performed at a constant frequency to find the linear regime of the elastic and viscous modulus. After that, the deformation is fixed and the frequency is scanned to find the modules.

The elastic or storage modulus (G') describes the strength of the material's elastic response, that is, the material's capacity to store energy; it represents the solid-like behavior of the material. Considering the same sample, as the elastic modulus decreases, the resistance to deformation and hardness also decrease. The viscous or loss modulus (G'') determines the fluid's ability to dissipate energy through the form of heat; it represents the viscous-like behavior of the material (Costa C. M.).

The principle of this method is:

- Initially, before gel formation, the sample exhibits the behavior of a liquid with $G'' > G'$.
- At the transition point, the gel starts to solidify.

- With $G' > G''$, the sample increasingly exhibits a gel-like behavior and, eventually, a solid-like behavior. Gelation time is considered the instant at which the elastic modulus is higher than the viscous modulus.

In Figure 6, we can see the results obtained for each concentration of sodium silicate. All of them have the elastic modulus surpassing the viscous modulus, indicating gel formation.

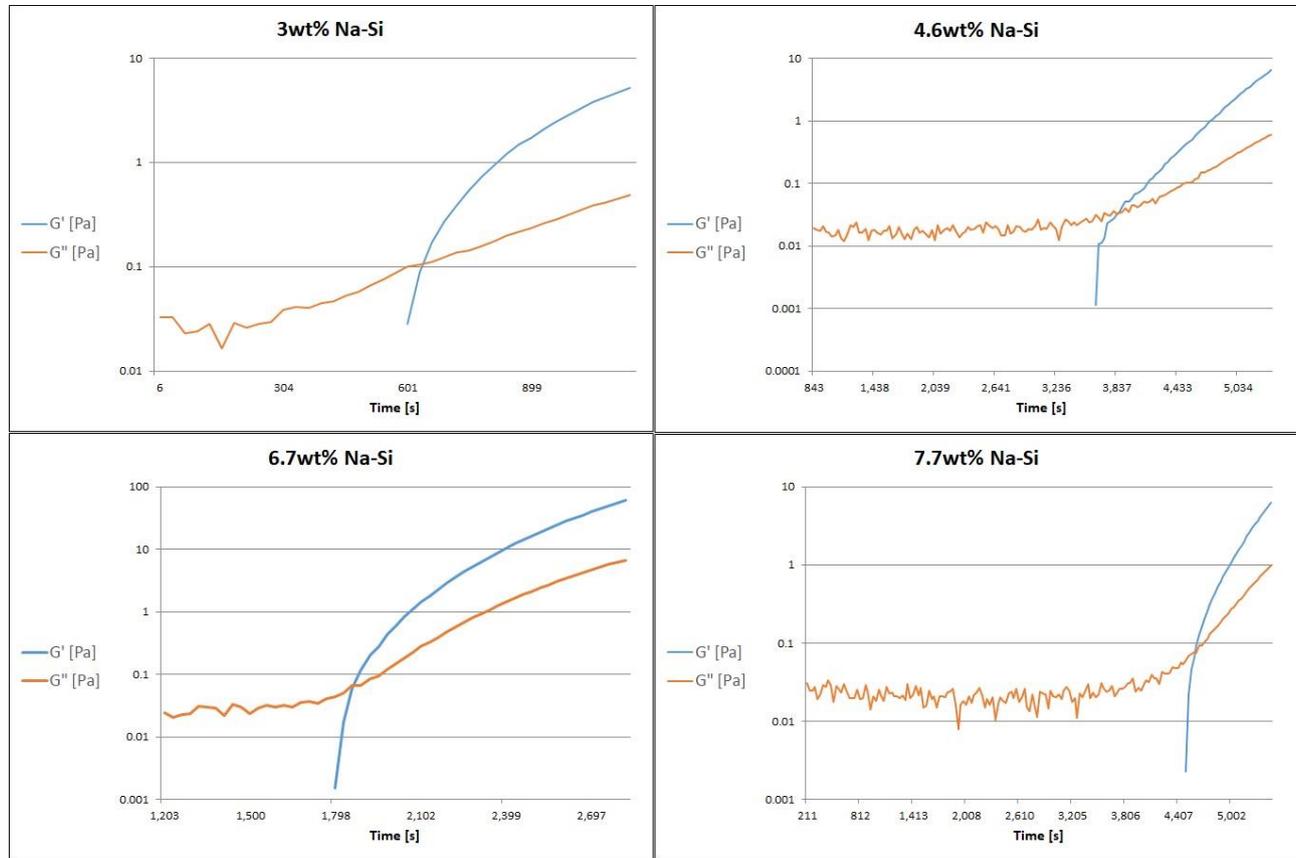


Figure 6: Oscillatory tests for each Na-Si sample

Table 4 shows the times it takes for the viscous and elastic modulus to cross, for each concentration of sodium silicate; and the time the viscosity starts to increase in the tests depicted in the previous section (inflection point), considering the samples with lower concentration of HCl. We can observe that the samples show approximate gelation times comparing the two tests, the least close being the 4.6wt% concentration. This indicates that the simple viscosity test can be used to compare the gelation time of different samples.

Table 4: Relation between sodium silicate concentration and gelling time for oscillatory and viscosity tests

Na-Si [wt%]	Time [s] (Oscillatory test)	Time [s] (Viscosity test)
3.0	632	870
4.6	3837	4900
6.7	1860	2130
7.7	4556	4680

There is a limitation in the manufacture of microcapsules, as it is difficult to encapsulate hydrochloric acid and maintain the structure, given its corrosive properties. Therefore, the sample chosen for the tests in the next section was the concentration 3wt% Na-Si, which requires a smaller amount of hydrochloric acid to gel and, consequently, a smaller amount of microcapsules.

3. TESTS USING MICROCAPSULES

A liquid formulation using microcapsules (Figure 7) aims to control the release time of the activating agent and, consequently, improve control over the beginning of the gelation process.

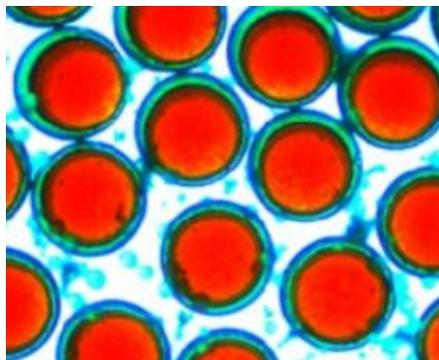


Figure 7: microcapsules with medium diameter of 500 μ m seen from microscope. Source: (*do Nascimento, et al., 2017*)

Osmosis is the passage of solvent through a semi-permeable membrane into a more concentrated medium. Osmotic pressure, on the other hand, is the pressure that must be exerted on the higher concentration solution to prevent osmosis from occurring. When there is an osmotic pressure gradient, the solvent passes to the more concentrated medium, in order to balance the pressure. This is the mechanism used to release the inner contents of the microcapsules in this work: when exposed to an osmotic pressure gradient in a hypotonic medium, they absorb the solvent and swell until they break their envelope.

Microcapsules are composed of a core and a shell; inside there is the activating agent and, when coming into contact with the sodium silicate solution, the capsules break by osmotic pressure and release the HCl to start the gelation process. The scheme is illustrated in Figure 8. In this way, the start of the gelation process can be controlled by the characteristics of the capsules, which will determine the time of membrane rupture when they are exposed to a certain difference in osmotic pressure.

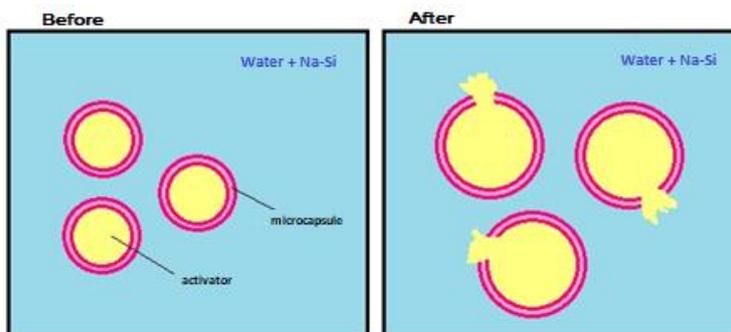


Figure 8: Illustration of Na-Si gelation through microcapsules

To produce monodisperse PDMS microcapsules, a double emulsion glass capillary device was used, consisting of a square outer capillary, a tapered injection capillary and a round collection capillary, as shown in Figure 9. The conical injection capillary and the round collection capillaries are inserted from opposite ends into the square capillary and coaxially aligned within the square, as shown schematically in Figure 9. The geometry of the double emulsion glass capillary device is kept constant while the fluid flow rates are independently varied and controlled through the use of syringe pumps coupled to the device by tubing. The internal aqueous phase composed of hydrochloric acid dyed with methylene blue is pumped through the round conical capillary, while the intermediate phase, PDMS, co-flows in the space between the square and round conical capillary. The external fluid, a 10% PVA solution, flows in the opposite direction in the space between the square capillary and the blunt-ended round capillary to focus the fluids and allow the formation of microcapsules (*do Nascimento, et al., 2017*).

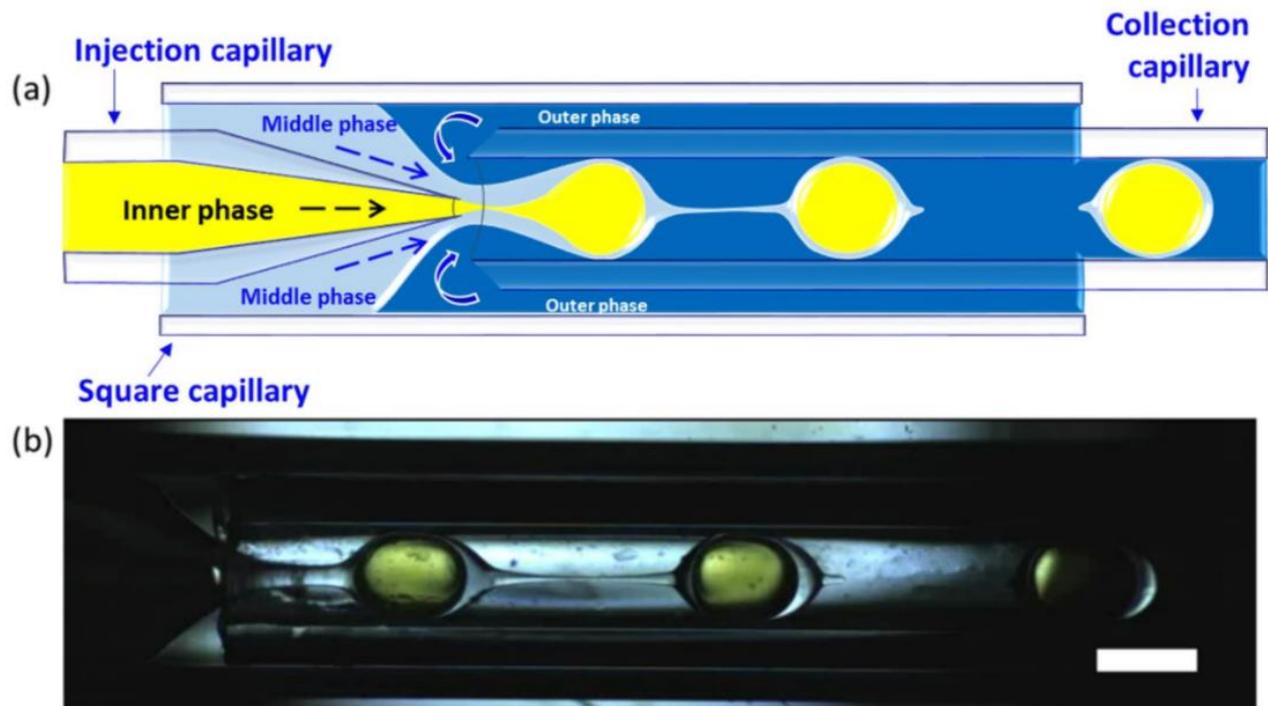


Figure 9: (a) Glass capillary device used to produce monodisperse PDMS microcapsules and (b) Image of double emulsion production process. Source: (do Nascimento, et al., 2017)

In the tests, a sample of sodium silicate with a concentration of 3wt% was used. The microcapsules are approximately 550 μm in diameter before the start of the experiment; and are suspended in high concentration sucrose to minimize the osmotic pressure gradient and prevent them from absorbing or releasing the contents before contact with sodium silicate. They were manufactured in a 10:1 ratio of PDMS and crosslinking agent.

The microcapsules are collected in several vials, and correspond to approximately 0.35% of the total volume. To use them in the experiment, it is necessary to concentrate them in the same vial. A syringe pump with a plastic tube is used, which extracts the sucrose at a rate of 5ml/min. The vial with all microcapsules concentrated in a single vial is shown in Figure 10.

One of the main objectives of this work is the proof of concept that the start of the gelation process can be delayed for a long time if the rupture time of the capsules is much longer than the gelation time. It is also important to check that the gelation process takes place after the capsules break.



Figure 10: Vial containing all microcapsules, after sucrose removal

3.1 Viscosity as a function of time

With the microcapsules already concentrated, the sodium silicate solution is added to start the viscosity test as a function of time in the rheometer, at a shear rate of $10s^{-1}$. This test was repeated at given time intervals.

In a first experiment, the sample was left under magnetic stirring at 360rpm, which was paused for five minutes before starting each viscosity test. A small 0.62ml sample was taken for each measurement. Eventually, the microcapsules agglomerated and formed the gel only around this cluster, as shown in Figure 11.

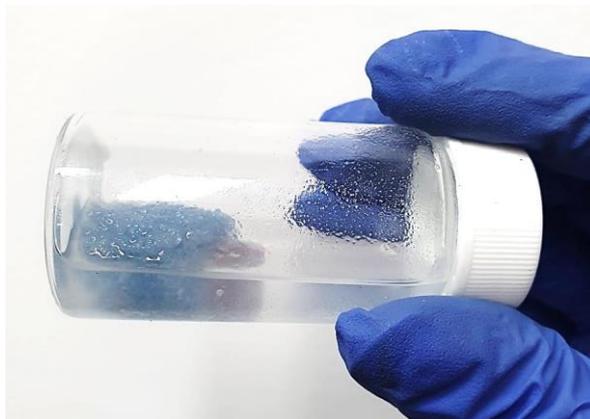


Figure 11: Cluster formed by microcapsules and gel

The viscosity as a function of time test showed that there was gelation and that it was delayed with the use of microcapsules in comparison with the results obtained in section 2, in which the HCl was added directly to the sample. As we can see in Figure 12, the viscosity started to increase between 50 and 60 hours of experiment, reaching the high values presented in Figure 4.

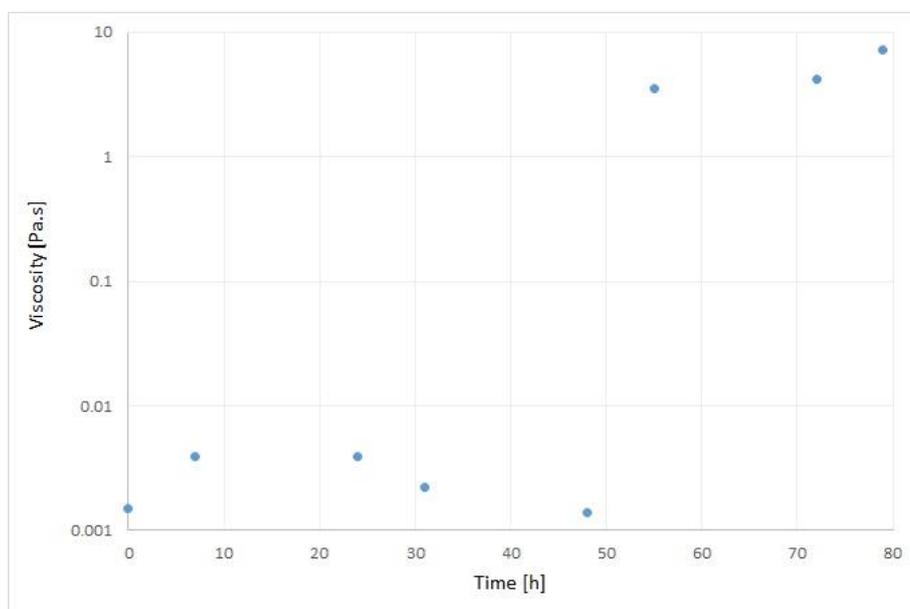


Figure 12: Viscosity test for Na-Si gelation through microcapsules

3.2 Conceptual gelation test with microcapsules

In order to avoid cluster formation, a second experiment was carried out in which, for 10 days, the Na-Si solution with the microcapsules was kept under magnetic stirring, at 360rpm, throughout the entire time. After five days in which the sample remained static (total elapsed time of 15 days), it was observed that the entire sample became gel, as shown

in Figure 13. Therefore, the hydrochloric acid was released by the microcapsules and reacted with the silicate sodium, causing the formation of gel. However, due to the constant movement of the magnetic stirring, it is not possible to notice an increase in viscosity in the tests performed on the rheometer. This is because the gel cannot form when shaken, it is necessary for the sample to remain static for transformation to occur.



Figure 13: Formed gel from microcapsule test

These preliminary results show that the microcapsules have great application capacity to control the release of content in the formation of sodium silicate gel, as they delay the beginning of the gelation process. However, further studies and tests are still needed to control the release time of the activating agent.

It is important to highlight that the break-up time can be controlled by the characteristics of the capsules used, such as the thickness of the membrane, material and composition of the polymeric membrane. This dependence is being currently studied.

4. CONCLUSIONS

A major problem faced by the industry in oil extraction is the presence of fractures in the reservoirs, which form preferential paths for the recovery fluid, leaving an accumulation of oil. An alternative to minimize this problem is the use of sodium silicate gel to block preferential paths of the recovery fluid, forcing it to go through unexplored pores. The main advantages of sodium silicate are the ease of extracting it in case of bad positioning and the fact that it is an environmentally “green” substance. However, for this solution to obtain the expected result, a very good control of the gelling mechanism and the positioning of the gel is necessary, in order to avoid problems.

In this work, the behavior and rheology of sodium silicate gel using hydrochloric acid as activator were studied. Different experiments were carried out to characterize the gel formation. In addition, conceptual tests of gelation with microcapsules were performed.

The results showed that the decrease in pH is an important trigger for gel transformation. The greater the amount of hydrochloric acid added to the sample, the faster the gelation occurs.

The transformation into gel was proven both in tests with rotational viscosity and in oscillatory tests. In all samples, the viscosity increases at least a hundred times; and the elastic modulus surpasses the viscous modulus, indicating that there is gel formation.

Preliminary tests with microcapsules demonstrate that it is possible to use them to control the release of content in the sodium silicate gelling process. It is still necessary to improve the experiments to fully evaluate the behavior of Na-Si samples with microcapsules. However, it was proven that the microcapsules release the content and can be used to delay gel formation from the Na-Si solution.

4.1 Next Steps

Studies with microcapsules containing hydrochloric acid for this type of application have already started. It is necessary to have good breakage control for them to be applicable in the field. Using this mechanism, it is likely to be easier to control the process of blocking the recovery fluid's preferential path through the sodium silicate gel.

It would be interesting to test microcapsules with a thinner shell than those used in this work. Thus, their breakage should be facilitated, as well as the release of the activating agent. This fabrication, however, is difficult to be carried out and demands greater specialization and control.

Another way to promote breakage is by manufacturing eccentric microcapsules, forcing the formation of a weak spot in the shell. Utada et. al. (Utada, et al., 2005) showed an example, shown in Figure 14.

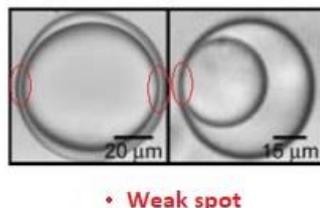


Figure 14: Eccentric microcapsules. Source: (Utada, et al., 2005)

In addition, it is possible to test the manufacture of microcapsules with a different ratio of PDMS and crosslinking agent, of 15:1, for example. Thus, the casing tends to be less rigid, which can favor its breakage. Or get the opposite effect, increasing its elasticity.

With the development of these and other tests, it will be possible to assess the applicability of microcapsules in controlling the release of the activating agent, resulting in a more controlled gelation process. Experiments are also important to determine the type of microcapsule to be used, as well as its characteristics. Thus, this mechanism can become an important ally in the oil industry and be used in the field, reducing the rate of residual oil in the reservoirs.

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