



Assessment of the influence of capillary internal diameter on scale inhibitors performance in DSL tests, especially at high temperatures with high TDS brines

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Abstract

The most cost-effective method to mitigate scaling – one of the major problems in Oil & Gas production – is the injection of scale inhibitors. Dynamic Scale Loop (DSL) is the technique used for assessing these chemicals efficiency in which is possible to access the Minimum Inhibitor Concentration (MIC). It consists in differential pressure measurements across a small diameter capillary tubing while two brines containing scaling constituents at a given flow rate injected. DSL method is very sensitive to the effect of capillary dimension, notably at high temperature in high scaling brines environment. The purpose of this study is to check the influence of capillary inner diameter in scale inhibitor performance. Three ID coils, 0.5, 0.75 and 1 mm, were used under three different temperatures, 120, 60 and 80°C. Test done at 120°C showed significant changes in scale inhibitor performance depending on capillary ID while testing at 80°C and 60°C no major changes were observed by varying capillary ID. Furthermore, the MIC was the same independently of testing coil. The results corroborates with the assumption that the capillary internal diameter might intensify the kinetics of salt formation reaction specially in high TDS brines at high temperature and so be an interfering factor in scale inhibitor performance.

Keywords

Scale Inhibitor; Dynamic Scale Loop; capillary ID.

Introduction

Inorganic scale deposition is one of the main problems related to the flow assurance in Oil & Gas Industry [1]. As a result of inorganic salt supersaturation that precipitates due to changes in operating conditions, i.e., temperature, pressure, and pH, dealing with its impacts represents a challenge since it can be very difficult to remove which ultimately leads to significant production loss [2]. The most viable and cost-effective method to mitigate scaling in the oil industry is the injection of chemicals known as scale inhibitors which aims to prevent the deposits formation. An effective scale inhibitor plays an important role on flow assurance in oil and gas production and, as such, to design a proper scale prevention treatment, the performance of these chemicals should be evaluated prior in bench-scale considering appropriate techniques [3]. The effectiveness and adequacy of these techniques employed to evaluate this class of chemicals is a key factor to be considered [4].

One of the most common and widely used techniques for assessing the efficiency of scale inhibitors as well as ranking its performance is the use of the well-known Dynamic Scale Loop systems. Through this method, it is possible to simulate conditions observed in field in order to not

only evaluate the scale inhibitor's performance but also study the scaling phenomena under pipeline and reservoir conditions [5,6,7].

The method's objective is to determine the MIC, defined as Minimum Inhibitor Concentration required to prevent a predefined increase in delta pressure over a certain time, and typically consists in a tubing block system in which measurements of differential pressure across a small diameter steel capillary tubing are done while two components of scaling brine are injected. The time pressure across testing capillary tubing increases and deviate from baseline gives the scaling time and the efficiency is measured by comparing time needed to block the tube in presence and absence of scale inhibitor, i.e. blank time [8].

Both thermodynamic and kinetic factors have influence on the precipitation process of inorganic salts – therefore on scale formation – and the so-called *threshold inhibitors* are considered kinetic inhibitors since it does not prevent the formation of scaling crystals, but it acts by delaying or retarding the rate of precipitation [9]. Threshold inhibitors affects the kinetics of the precipitation process and, depending on their chemistry, they can act through two mechanisms: (i) preventing nucleation or (ii) crystal growth of scaling species, allowing supersaturation without scale formation [10].

Considering the aforementioned mechanisms are time dependent, residence time is an important factor in defining the methodology to evaluate the efficiency of scale inhibitors. DSL method is very sensitive to the effect of flow rate and capillary tubing dimension, notably at high temperature in high scaling brines environment, which can directly impact residence time leading to misinterpretations of results [4,10]. Inhibitors that act in the nucleation phase may benefit more from short residence times, while those that prevent crystal growth may perform better at longer residence times. Failure to provide adequate conditions to perform the analysis may fail to consider the most relevant mechanism of the inhibitor.

Capillary tubing with very thin internal diameter has just a few seconds of residence time so as the reduction of ionic species while scale is formed decreases the saturation ratio it can possibly cause unequal distribution of deposited scale along the coil which has a significant impact on inhibitor performance. In addition, the tube internal diameter may accelerate the kinetics of the precipitation in capillary inlet [3] especially at high temperature and high salinity conditions that could lead to incorrect assessment of scale inhibitor MIC.

As such, this study presents and describes a series of DSL testing performed with a high salinity brine at different inner diameter (ID) capillary coils and at different temperatures in order to evaluate the influence of ID and reaction kinetics on scale inhibitors performance, with the purpose of a better representation of production reality also providing more adequate testing parameters for scale inhibitors evaluation in order to avoid misinterpretations of products efficiency, particularly at high stress scenarios.

Methodology

The methodology for this work included software simulations to predict the scale potential and dynamic efficiency tests to evaluate the scale inhibitor performance in dynamic conditions also the influence of testing capillary inner diameter on reaction kinetics at different temperatures.

Scale Soft Pitzer software, developed by the Brine Chemistry Consortium at Rice University (M. Thomson's and A. Khan's group) were used to perform the simulations.

Dynamic Scale Rig model H400 from Techbox Systems were used to perform dynamic tube blocking test. The instrument can reach up to 6,000 psi and is equipped with two double pistons pumps (Knauer Azura P4.1S), one used for cationic brine and other for anionic or "inhibited anionic" brine and the cleaning solutions; as part of the equipped there is also a Memmert oven model UF55Plus, that is suitable for temperature up to 300°C. During testing, equipment records automatic data of differential pressure, measured by a pressure transducer, between the inlet and outlet of a stainless-steel capillary coil.

Experimental Procedure

Preparation of synthetic brines

To perform laboratory test, synthetic brines with similar composition of the scenario to be evaluated were prepared. Brine composition considered in this study is shown in Tab. 1.

Table 1. Brine composition.

Species	Composition (mg/L)
Na ¹⁺	78,600
K ¹⁺	4,700
Mg ²⁺	1,200
Ca ²⁺	6,160
Sr ²⁺	3,000
Ba ²⁺	44
Cl ¹⁻	138,900
Br ¹⁻	750
HCO ₃ ¹⁻	1,550
SO ₄ ²⁻	90
CH ₃ COO ¹⁻	940
pH C⁺	5.20 +/- 0.20
pH A⁻	7.15 +/- 0.05

Both cationic and anionic brines were prepared by adding all the required number of salts to DI water. BaCl₂*2H₂O, CaCl₂*2H₂O, NaCl, KCl, SrCl₂*6H₂O and MgCl₂*6H₂O (ACS Reagent, Sigma Aldrich) were used for synthetic cationic brine. For synthetic anionic brine NaCl, NaHCO₃, NaSO₄, CH₃COONa and KBr (ACS Reagent, Sigma Aldrich) were used. Brines were left under stirring overnight, then filtered with a 0,45 µm pore size membrane. NaHCO₃ were added to the anionic brine prior each test. Both brines had their pH adjusted shortly before testing began. The pH of anionic brine was buffered by bubbling a mixture of CO₂ and N₂ while to buffer the pH of cationic brine HCl and NaOH solutions were used.

Scale inhibitor

The scale inhibitor (SI) considered in this study was a product based on a salt solution of Polyamino Polyether Methylene Phosphonic Acid (PAPEMP) from Italmatch Chemicals. The product shows great compatibility with high TDS, high pH brines and winterizing agents. Highly thermal stable with outstanding performance against calcium carbonate and calcium sulfate scale.

Software Simulations

Saturation Indexes (SI) and mass of precipitate to re-establish the equilibrium (Deposit rate) considering brine composition under all testing conditions of temperature and pressure were calculated through Scale Soft Pitzer software. It is important to highlight that the predictions are based on thermodynamics assumptions.

Dynamic Scale Loop test.

A series of dynamic tube blocking tests were performed to assess the scale inhibitor performance in three different conditions of

temperature and considering three different internal diameter capillary coils. To perform this test, anionic and cationic brines were pumped separately at 1:1 ratio through two 6-m-long pre-heating coils each and then combined by a union tee in a 1/16" OD 1-meter stainless-steel coil. Three ID were considered: 0.5 mm, 0.75 mm and 1 mm. Temperature and pressure were set up based on field conditions. Table 2 summarizes all testing conditions.

Table 2. DSL testing conditions.

Parameter	Conditions
Temperature	120, 80, 60°C
Pressure	8 bar
Coil ID/length	0.50, 0.75, 1.00 mm/1000 mm
Flow rate	10 mL/min
Testing time	120 min

After each test, 10% acetic acid solution, 5% alkaline EDTA solution and DI water were used to clean the coil. In each experiment, a successful test was when the pressure does not surpass the target threshold of 0.5 psi after 120 minutes. The experiment is designed as a single dosage test, in which only cationic and inhibited anionic brine were used. When the testing dosage was unsuccessful – which means that the threshold differential pressure value is achieved – a new inhibited anionic brine with a different dosage was prepared.

Results and Discussion

In order to know the severity and types of scales predicted, simulations were performed, and the data obtained considering brine composition and scenario's pressure in all three temperatures are summarized in Tab. 3.

Table 3. SI and Deposit rate predicted.

Scale	SI/Deposit rate at 120°C
Calcite	2.98 / 1,119
Celestite	0.33 / 90
Barite	0.18 / 20
Scale	SI/Deposit rate at 80°C
Calcite	2.78 / 1,065
Celestite	0.25 / 74
Barite	0.49 / 45
Scale	SI/Deposit rate at 60°C
Calcite	2.69 / 1,033
Celestite	0.21 / 64
Barite	0.69 / 55

The simulations show that the main concern is calcite formation with high SI and deposit rates expected in all three temperatures. Sulfates scales are predicted as well but with less intensity. To assess the efficiency of scale inhibitor and evaluate the influence of capillary inner diameter, DSL were performed considering the three ID in all three temperatures. The aim for these tests is to observe, while temperature is reduced from 120°C to 60°C, the reduction of kinetics influence in precipitation, therefore, being possible to see if the

capillary inner dimension is interfering in inhibitor performance since at higher temperatures the kinetic effects of precipitation – not necessarily of scale formation – are more prominent and so the capillary tubing would be a determining factor of scale performance rather than the scale phenomenon itself, which would be beyond the scope of field reality.

Besides the kinetic factor, it should be noticed that the capillary internal diameter also influences flux velocity. As grater as the internal diameter, flux velocity is reduced, leading to longer residence time for higher ID. Since threshold scale inhibitor mechanisms are time dependent, it is expected that for higher ID the efficiency of scale inhibitor would be more appropriately evaluated considering a lower brine flow velocity resulted from the mixture of cationic and anionic brines while passing through testing coil.

For each temperature, except for 120°C, scale inhibitor MIC were determined. The DSL results performed at 120°C considering the three IDs are shown in Fig. (1). Considering the maximum dosage of 500 mg/L, the MIC could only be determined in the 1 mm ID coil which was found to be 300 mg/L. The tests showed that at 120°C significant changes in scale inhibitor performance were experimentally observed depending on capillary ID. This fact corroborates the assumption that the capillary internal diameter might intensify the kinetics of salt formation reaction which already is greater at high temperatures. The capillary tubing ID interference might prevent a more adequate scenario/condition to evaluate products performance against *scale* itself since the speeded kinetics can lead to an accelerated amount of solid formation that stave off the scale inhibitor of its true capacity.

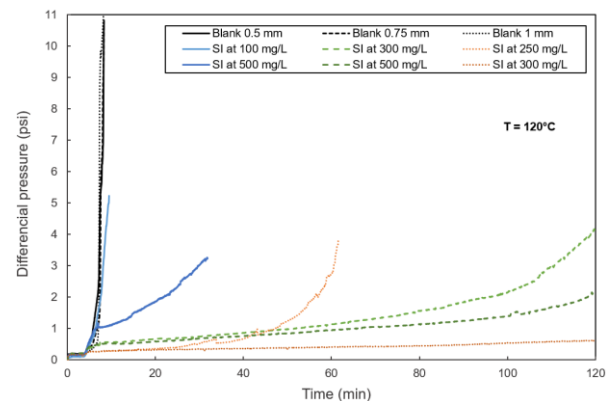


Figure 1. DSL tests at 120°C.

For the other two temperatures evaluated, 80 and 60°C, the MIC determined were 50 and 20 mg/L respectively and independently of capillary ID. Taking into account that with decrease in temperature there is also decrease in kinetics effects, seeing that while changing the coil no significant changes were observed in scale performance also the MIC were found to be the same under each temperature, it is possible to infer

that there is less or no interference of capillary ID in products behavior when reaction kinetics are reduced, so the capillary does not act as interferent in scale inhibitors performance.

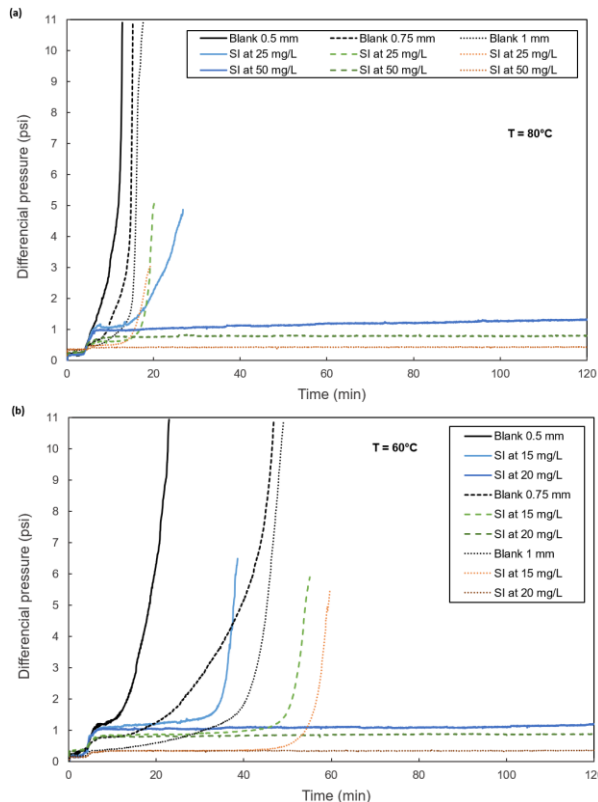


Figure 2. DSL tests at (a) 80°C e (b) 60°C.

All above mentioned and results obtained from DSL testing implies that it is very important to consider the influence of capillary internal diameter on kinetics effects thus its interference in scale inhibitor performance in higher TDS brines especially at higher temperatures, in which this phenomenon is intensified. It was observed that under these conditions as thinner as the ID, the greater is its influences in products behavior.

Conclusions

It was shown in this work that the inner coil ID can be an interference factor for scale inhibitor performance since it influences flow velocity, therefore residence time after mixture of cationic and anionic brines, and its effects on kinetics of precipitation specially at higher temperatures. Thereby, an appropriate analysis of product's performance against scale formation is not possible without considering this influence. Testing procedures must attempt to mimic field conditions as much as possible to drive a true selection of efficient products also determine appropriate MIC. Thus, it is very important to select a proper way to access the efficiency of scale inhibitor against scale formation considering suitable inner ID coils, especially at high TDS and high temperature conditions, therewith reducing associated uncertainties of upscaling from laboratory to field,

avoiding wrong assessment of real treatment capabilities of scale inhibitors.

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Responsibility Notice

The authors are the only responsible for the paper content.

References

- [1] Li, J. et al; *J. of Disp. Scien. and Tech.* 38:661–670, 2017.
- [2] Kamal, M. S. et al; *J. of Petrol. Scien. and Eng.* 171:127–139, 2018.
- [3] Olujide, S. S. et al; *J. of Petrol. Scien. and Eng.* 182:106347, 2019.
- [4] Graham, G. M. et al. *International Symposium on Oilfield Scale*, SPE-74679, Aberdeen, United Kingdom, 2002.
- [5] Al Helal, A. et al; *J. of Petrol. Scien. and Eng.* 179:1063–1077, 2019.
- [6] Mohamed, F. M. *Ind. & Eng. Chem. Res.* 60(33):12175–12188, 2021.
- [7] Silva, C. M. F. da, et al. *Petroleum Science and Technology*, 1–19, 2024.
- [8] Amjad, Z. *Mineral Scales in Biological and Industrial Systems*. Ed. CRC Press, Flórida, Estado Unidos, 422 p, 2014.
- [9] Senthilmurugan, B. et al. *International Journal of Petroleum Science and Technology.* 13(1): 21–38, 2019.