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EXPERIMENTAL INVESTIGATION OF THE EFFECT OF SCALE INHIBITORS ON THE PRESSURE DROP AND ON THE DEPOSITION INSIDE HYDROCYCLONES

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Abstract. *Scaling is one of the problems faced by the oil industry and can occur in valves, pipelines and equipment, leading to increased time and costs for cleaning and maintenance of the production line. This study aims to evaluate the efficiency of a chemical inhibitor in mitigating the calcium carbonate ($CaCO_3$) scaling process in hydrocyclones. Progressive cavity pumps were used to mix sodium bicarbonate and calcium chloride solutions in an one-inch diameter (OD) stainless steel pipe that fed an encapsulated hydrocyclone. Parameters such as flow rate and pressure were monitored throughout the test period, and liquid samples were collected for pH, conductivity, temperature, size and quantity analysis of $CaCO_3$ particles. The deposited mass inside the hydrocyclone and in the tube sections distributed along the experimental bench was characterized by weighing. The crystal structure of calcium carbonate was analyzed by scanning electron microscopy (SEM). The main contribution of this research is the evaluation of the minimum concentration inhibitor required so that, under real operating conditions, mass deposition is not observed in the platforms' hydrocyclones.*

Keywords: *scaling, calcium carbonate, chemical inhibitor, hydrocyclones.*

1. INTRODUCTION

Although not exclusive to the petrochemical industry, inorganic scaling causes several problems in oil recovery processes. This happens mainly due to partial or total obstruction of pipelines, which increases the frequency and time required for equipment maintenance and cleaning, resulting in a significant drop in the production process (Li *et al.*, 2022; Matos and Atloé, 2019; Reis *et al.*, 2011).

For oil recovery, a mixture of formation water and injection water (typically seawater, used to maintain satisfactory reservoir pressure) is used. While the formation water is rich in dissolved chemical elements such as calcium, barium, and strontium, and the sea water is primarily composed of chlorides, bicarbonates, and sulfates. When these two waters are mixed, the formation of inorganic salts occurs, which deposit on the pipelines (Motta *et al.*, 2013; Reis *et al.*, 2011).

Therefore, mitigating or inhibiting scaling phenomena is crucial for the industry. One of the most well-known, efficient, and cost-effective methods for doing so is the use of chemical inhibitors (Fernandes *et al.*, 2021). In general, these products contain functional groups with chelating effects, responsible for capturing or adsorbing ions in solution and acting on the growth and nucleation stages of crystalline structures, thus disaggregating and breaking down deposited molecules, as well as reducing the size and delaying crystal growth (Guicai *et al.*, 2007; Li *et al.*, 2022).

With simple manufacturing, maintenance, and operation, hydrocyclones are separation equipment widely used by the petrochemical industry in the last 60 years. Its main application is to clean the produced water from petroleum residues (Vieira *et al.*, 2005). Hydrocyclones provide fast online separation due to the centrifugal force imposed by rotational flow. These devices separate solid, liquid, or gas mixtures based on density differences (Coelho, Neto and Medronho, 2011; Jank *et al.*, 2017).

The present study aims to evaluate the performance of chemical inhibitors to mitigate and prevent calcium carbonate ($CaCO_3$) scaling under rotational and turbulent flow conditions in hydrocyclones.

2. EXPERIMENTAL METHODS

The main purpose of the experiments was to determine the effectiveness of the chemical inhibitor used in preventing calcium carbonate scaling in a hydrocyclone operating under real field conditions. This study was conducted at the Interdisciplinary Center for Fluid Dynamics (NIDF) at the Federal University of Rio de Janeiro.

To perform the experiments, two tanks of 2700 l with different solutions are prepared, stored and monitored until stabilization. One tank is prepared with a concentration of 0.00735 kg/l of $CaCl_2.2H_2O$, the other with 0.0126 kg/l of $NaHCO_3$, both tanks are monitored during a period of at least 5 days for stabilization, being continuously stirred by mixers to ensure homogenization of each solution and that the balance of CO_2 with the environment is quickly reached. In addition, the two solutions are analyzed daily, measurements of pH, conductivity and temperature are performed.

Each solution, after stabilization, is pumped by two progressive cavitation pumps (PCP) to the experimental setup during the experiment. This mixture is then transported, by a 1" stainless steel pipe, to a hydrocyclone prototype stored in a capsule. The flow rate split is controlled by a gate valve in the underflow and a needle valve in the overflow. Fig. 1 presents a schematic diagram of the setup.

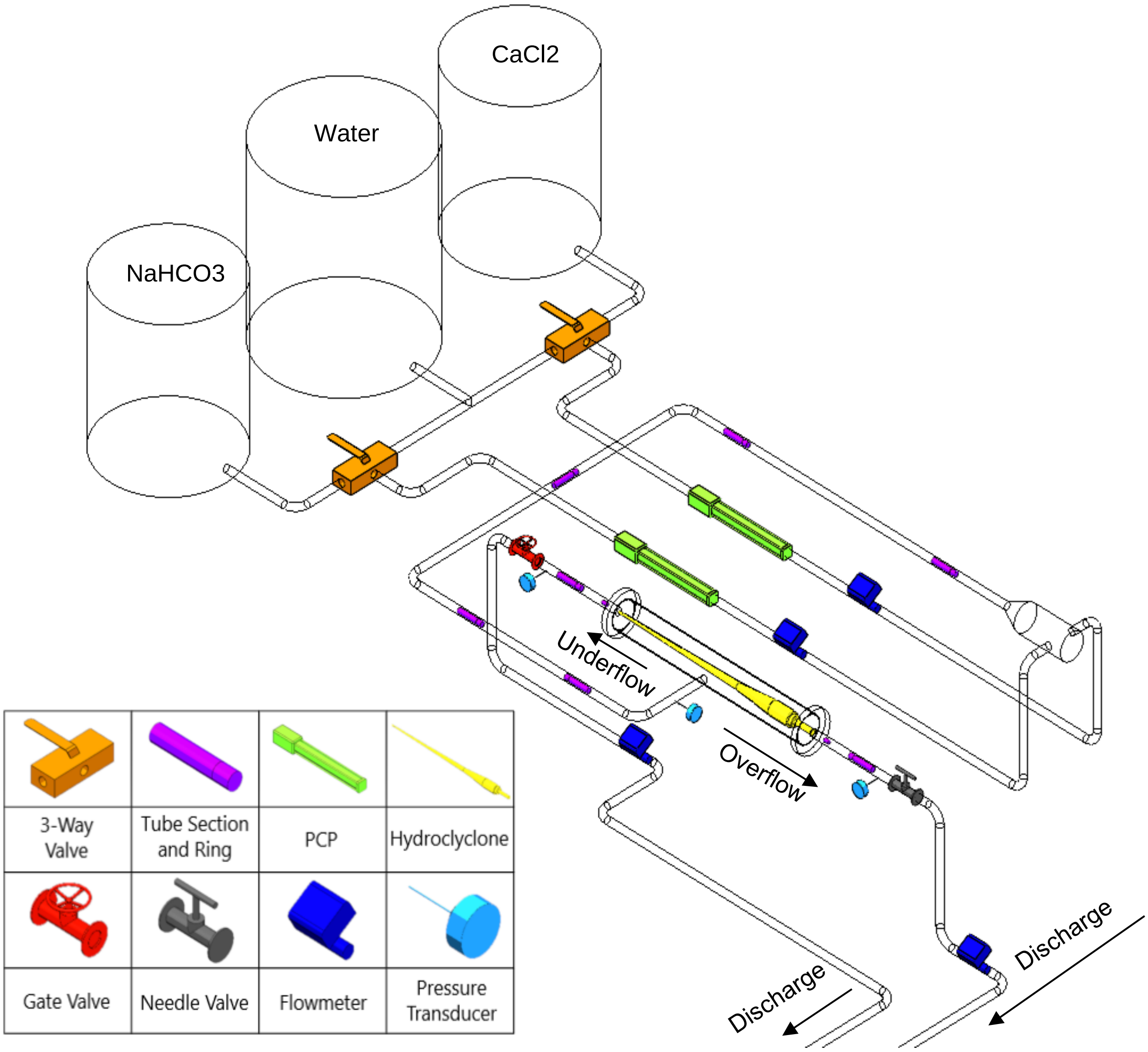

Figure 1. Schematic diagram of the experimental setup.

During the experiment, pressure and flow rate data are collected and monitored at specific points on the setup. The flow rate is monitored in each of the PCPs and in each of the outlets of the hydrocyclone, by four flow meters, while the pressure is monitored at the inlet and outlets of the capsule by three pressure transducers.

The conditions of the experiment were chosen to replicate the real field operating conditions provided by Petrobras. Each solution is introduced to the setup, through the PCPs, at a flow rate of 1000 l/h each, a split of 2% is imposed in the overflow, such that at a flow rate of 40 l/h is observed, these are kept constant throughout the experiment through the manipulation of the pump inverter and the needle valve. The initial pressure in the inlet is 8 bar. All these parameters are reached using a water reservoir, before the introduction of the solutions. All experiments were carried out at an ambient temperature.

Experiments are performed to determine the inhibition efficiency of chemical inhibitor. A number of tests are performed, including blank tests, without any inhibition method. The addition of the chemical inhibitor is carried out in the $NaHCO_3$ tank, 24 hours before the beginning of each test. During the experiment, it is possible to monitor the effect of inhibition by observing the pressure on the system; if scaling occurs, the pressure rises constantly, otherwise it remains constant. The chemical inhibitor used has Polyamino Polyether Methylene Phosphonic Acid (PAPEMP) as active material.

Additional tests were performed at CENPES/PETROBRAS to determine the MIC in a capillary tube. The results obtained with the capillary tests served as an initial parameter for the concentration in the real scale experiments. The concentrations tested in the capillary tube were: blank (0 ppm), 50 ppm, 60 ppm, 100 ppm and 200 ppm.

For the tests carried out on the experimental bench, the chemical inhibitor concentrations shown in Table 1 were used.

Table 1. Experimental conditions.

Name of experiment	Tested Concentrations (ppm)
Blank	0
CI-100	100
CI-130	130
CI-140	140
CI-150	150
CI-200	200

The experiment is finished when a pressure of 20 bar is reached in the inlet, or upon depletion of the solution tanks. Just before the end of the test, liquid samples are collected at five different locations, three in the pipe before the hydrocyclone capsule and two after. These liquid samples are analyzed, determining number and size of particles, through the PAMAS particle counter and Mastersizer 3000, respectively. In addition, after the experiment, seven sets of tube sections and rings of 10 and 2 cm in length respectively, which were previously introduced to the experimental setup, represented in the schematic on Figure 1, are removed from the pipe, dried and weighed. The morphology of calcium carbonate deposition is also analyzed in the smaller tube sections (rings) by scanning electron microscopy (SEM). In addition to the tube sections, the hydrocyclone is also dried and weighed after the end of the experiment to determine the deposited mass and the deposition rate.

3. RESULTS

In this section the results obtained in all experiments are presented. Results will be presented for the capillary test performed, as well as for the Blank test and for tests with chemical inhibitor in the concentrations used (CI-100, CI-130, CI-140, CI-150 and CI-200).

First, tests were carried out on a pilot scale using a concentration of 200 ppm (results will be shown throughout the work), verifying that the inhibitor would be efficient at this concentration, without formation of calcium carbonate scaling. After these tests, a capillary test was performed to verify the minimum concentration at which inhibitor would be effective. Figure 2 shows the temporal evolution of the differential pressure obtained at the capillary (tube blocking) test. Based on these data, it was identified that the chemical inhibitor used would be efficient at a concentration of 100 ppm. Thus, experiments were carried out on the experimental bench at concentrations lower than 200 ppm to verify the minimum effective concentration to be used on the bench.

Figure 3 presents a comparative analysis of the temporal evolution of the inlet pressure (P_{inlet}) for the blank and chemical inhibitor tests at concentrations of 100, 130, 140, 150 e 200 ppm. Results show that the chemical inhibitor was effective in the concentrations of 150 and 200 ppm. It is also possible to see that at a concentration of 100 ppm the inhibitor did not perform well, since there was an increase in inlet pressure over time.

With the aim of finding out if the minimum effective concentration of the inhibitor for the experimental bench under study was 150 ppm, experiments were carried out using 130 and 140 ppm of chemical inhibitor. It was observed that at concentrations of 130 and 140 ppm the pressure signal does not behave like the Blank test nor like the tests carried out at effective concentrations of inhibitor. This leads to the conclusion that there is an increase in pressure and these concentrations are not effective.

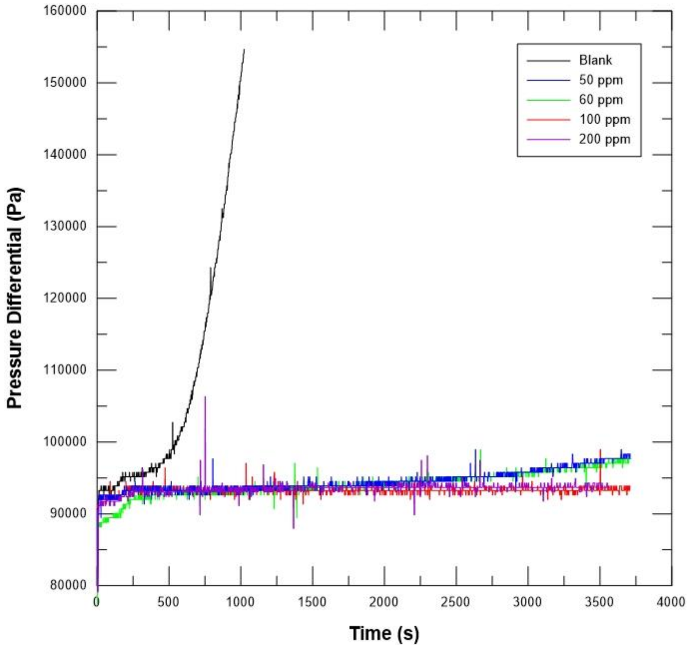

Figure 2. Differential pressure measured at the capillary test Blank and with chemical inhibitor at concentrations 50, 60, 100 and 200 ppm.

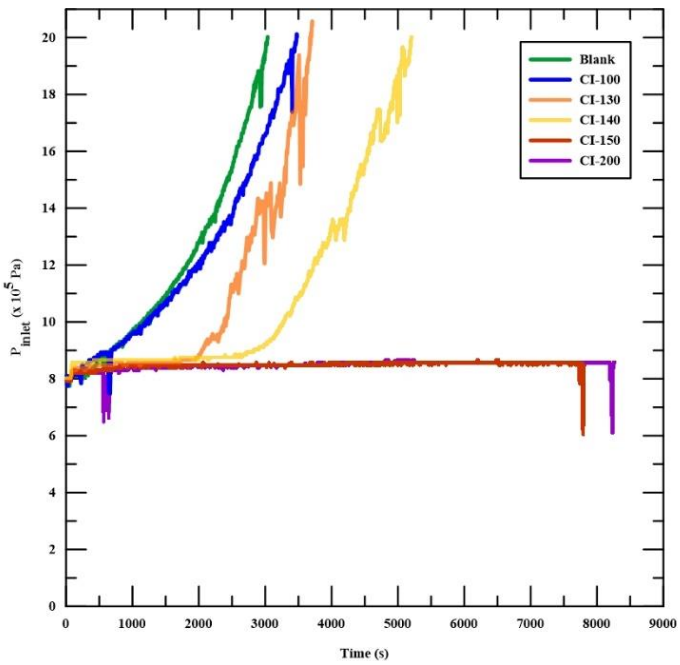

Figure 3. Temporal evolution of the inlet pressure (P_{inlet}) for Blank and chemical inhibitors tests at concentrations of 100, 130, 140, 150 and 200 ppm.

After the end of the experiments, the hydrocyclone is removed from the experimental bench and placed to dry for 48 hours. After this time, the hydrocyclone is weighed and the deposited mass of calcium carbonate inside is obtained from the difference in mass. Furthermore, with this deposited mass values, it is possible to calculate the deposition rate inside the hydrocyclone. Table 2 presents the deposited mass and deposition rate values inside the hydrocyclone.

Table 2. Values of deposited mass and deposition rate inside the hydrocyclone.

Test	Deposited Mass inside HC (x 10^{-3} Kg)	Deposition Rate (Kg/m^2.s)
Blank	27,09	1,47 x 10-4
CI-100	24,31	1,15 x 10-4
CI-130	13,89	6,13 x 10-5
CI-140	4,81	1,51 x 10-5
CI-150	0	0,00
CI-200	0,43	8,56 x 10-7

Through the deposition rate values, it is noticed that they are smaller or almost immeasurable for the experiments carried out with chemical inhibitor at concentrations of 150 and 200 ppm, confirming the effectiveness of the chemical inhibitor at these concentrations.

With the end of the experiment, the removable tube sections and rings of the experimental bench are also removed. They are dried, weighed and the deposited mass inside the tube sections is obtained from difference in mass. Figure 4 shows the deposited mass in tube sections.

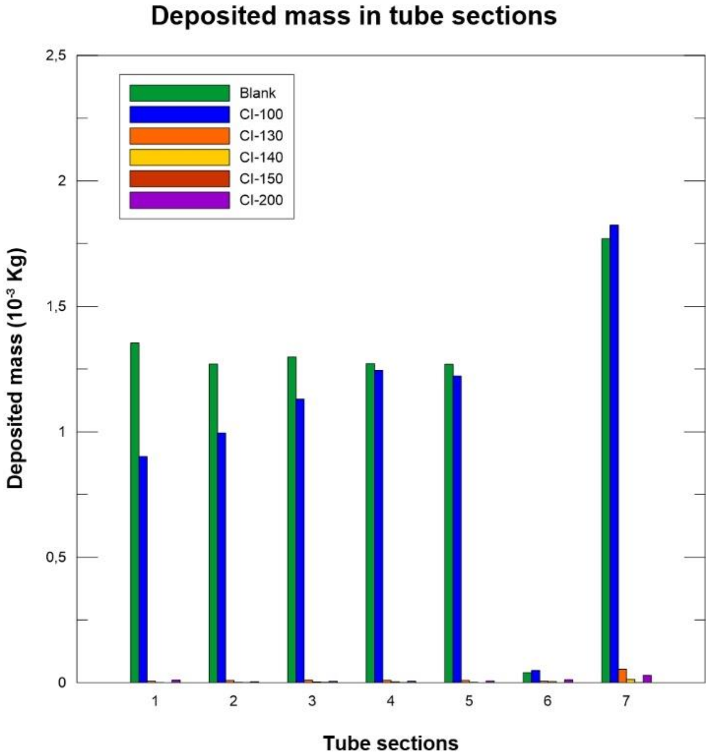

Figure 4. Deposited mass in tube sections for all experiments carried out in the experimental bench.

As Figure 4 shows, the masses in the tube sections follow the same behavior of the deposited mass inside the hydrocyclone. For the experiments carried out with concentrations of 150 and 200 ppm no significant values of the deposited mass were obtained.

With the rings removed from the bench, Scanning Electron Microscopy (SEM) analyses were performed to evaluate the crystal structure of the deposited solid material. The blank test shows the formation of calcite as a crystalline form of calcium carbonate. For the experiment carried out at a concentration of 100 ppm of chemical inhibitor, vaterite was found as a crystalline structure. Thus, the use of chemical inhibitor can alter the morphology of calcium carbonate crystals, forming a less stable crystalline structure than calcite. Figure 5.a shows the microscopy for the blank test (calcite) and Figure 5.b shows the crystalline form of vaterite, removed from the test with chemical inhibitor.

As previously mentioned, all experiments were carried out at an ambient temperature. If the experiments had been carried out at different temperatures, higher than ambient temperature, it would have been possible to find a greater amount of deposited mass, in addition to different crystalline forms. This could occur, since the solubility of calcium carbonate in water is lower at higher temperatures.

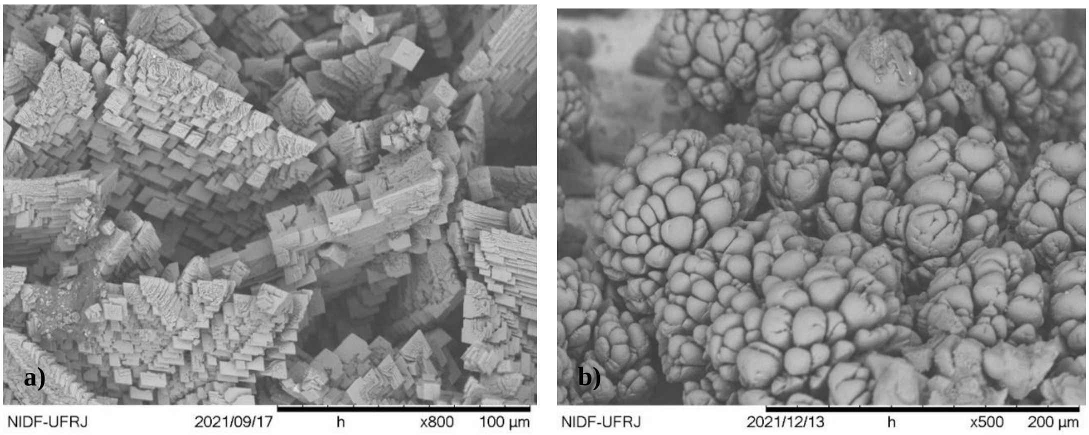

Figure 5. Scanning electron microscopy images of the Blank test (a) and the experiment with 200 ppm of chemical inhibitor (b).

4. CONCLUSIONS

Results show that the chemical inhibitor is efficient for some concentrations, with a minimum effective concentration of 150 ppm. This can be confirmed through the pressure signal, which was maintained approximately constant throughout the experiment, with no calcium carbonate scaling. In addition, the results of deposited mass inside the hydrocyclone, deposition rate and deposited mass in tube sections also confirm the conclusion that the inhibitor is efficient at a concentration of 150 ppm, since in the Blank test and the test with 100 ppm of chemical inhibitor there is deposited mass and at a concentration of 150 ppm there is not.

Another important factor is that the minimum effective concentration determined by the capillary test, of 100 ppm, is not the same as determined on the experimental bench, showing that the concentration determined by the capillary test may not be applicable to hydrocyclones.

5. ACKNOWLEDGEMENTS

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