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Scale Inhibition by Ultrasonic Reactor Operating in Pipelines

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Abstract: ***Calcium carbonate scaling can lead to several problems in the oil and gas industry, reducing pipe flow and deteriorating thermal processes. This work investigates the mechanism of scale inhibition using ultrasonic technology. An ultrasonic device designed to minimize the adhesion of calcium carbonate crystals in pipelines is detailed. The device equipped with three high-power ultrasonic transducers (60 W x 40 kHz) was used to stimulate calcium carbonate precipitation in a hydraulic system specifically assembled to study the calcium carbonate scaling by simultaneous injection of $CaCl_2 \cdot 2H_2O$ and $NaHCO_3$. A transparent plastic hosepipe 5 m long was used as a proof-pipe enabling visual observation. Pressure drop and crystal morphology were evaluated. Ultrasonic field evaluation tests inside the developed device showed strong cavitation activity and significant erosion on aluminium foils. After 140 minutes of continuous flow through the pipe under US irradiation, volumetric flow rate remained stable. A reduction in hydraulic resistance of 42% was found, if compared to the test condition without any scaling protection. The morphology of the crystals evaluated by scanning electron microscopy showed that crystals became smaller and more rounded.***

Keywords: ultrasonic reactor, scaling, calcium carbonate, crystals precipitation.

I. INTRODUCTION

Salt scaling such as $CaCO_3$, $CaSO_4$ and $SrSO_4$ is a major problem for processing industries, imposing high costs, especially in petroleum sector due to production efficiency reduction, as well as costs in maintenance services on pipelines and equipment such as heat exchangers, and evaporating coolers[1]-[3]. Calcium carbonate is the most common scaling which as supersaturated conditions tends to deposit continuously on the inner pipe surface, decreasing flow and causing localized corrosion [4]-[6].

Several methods have been used to remove carbonate deposits. Although chemical methods can be successfully used to remove scaling, they are costly, cause severe environmental damage, and need to be periodically reapplied [7], [8]. Some studies have focused on the use of physical devices to prevent scaling, such as electrical, magnetic, and ultrasonic methods [9], [10].

Ultrasound (US) is a non-destructive cleaning and anti-scaling method used in manufacturing industries, combining good energy performance and very low environmental impact. High power ultrasonic transducers have already been used to inhibit crystal growth on metal surfaces in industrial applications such as water treatment, boilers, pipes and heat exchangers [11],[12].

Although the mechanisms and anti-scaling performance are not yet fully understood, US applied under low frequency (20 to 100 kHz) can generate cavitation in liquids. Some of the generated bubbles collapse asymmetrically close to the solid surface inducing shear forces resulting in crystal agglomerate rupture, as well as preventing the aggregation of new crystals on a solid surface, even in a supersaturated solution [13]-[18]. Aiping et al.[14] performed a theoretical simulation evaluating the influence of ultrasonic wave parameters on cleaning efficiency of fouled surfaces. The authors found that an increase in parameters such as the velocity and viscosity of the liquid attenuates sound waves. They also found that increasing the ultrasonic frequency implies higher attenuation.

Delacour et al. [15] designed a flow reactor and compared simulation data with experimental results. The reactor consisted of six Langevin-type variable frequency transducers. Acoustic pressure distribution and transducer configurations were evaluated, studying size distribution of barium sulfate particles at 40 kHz. Peak acoustic pressures were observed near the tubular reactor, along with a reduction in particle size, which minimized clogging.

Geng et al. [5] carried out an experimental study evaluating descaling performance of US at 20 kHz, powered by 80 W transducer, on the water treatment on the heat transfer process.

The results showed that US altered morphology of formed aragonite crystals, pointing out that the technique used is effective in heat transfer applications with high hardness water. Li et al. [16] developed a method to determine the anti-scaling efficiency capacity of calcium carbonate in solutions under several ultrasonic treatment conditions. The authors found a maximum value of 81.1 % anti-scaling efficiency using US at 28 kHz. Liu et al. [18] tested the use of ultrasonic irradiation as an anti-scaling alternative in the milk pasteurization industry. A transducer working at 40 kHz was attached to the pipe wall. They found the US anti-scaling effect was reduced when the flow rate and temperature increased or when the device operated in curved sections due to reflection effects.
Mazue et al. [13] evaluated the effectiveness of an automated cleaning system, for immersed boats, using mobile probes containing three US low-frequency transducers. The authors evaluated the influence of parameters such as probe position, speed and power. Good surface cleaning efficiency was found, and no wave destructive interaction was detected between two or more probes, even when mounted close to each other. Moilanen et al. [19] tested a clamp-on high power ultrasound device in a tubular heat exchanger. Borescope images taken after 12 days of operation showed that the heat exchanger surfaces remained clean, allowing on-site fouling mitigation. Banakar et al. [20] investigated the application of US (33k Hz) in a double pipe heat exchanger aiming at reducing scale formation and in-situ cleaning. Reduction in average fouling resistance by 43.74% was found when the flow rate was 600 mL/min.
As discussed earlier, US is a mechanical method to keep inner surface of pipelines free of deposits, ensuring appropriate flow rate, improving heat transfer in thermal processes, as well as maintaining the purity of the pumped fluid. This work aimed to evaluate the mechanism of scale inhibition using ultrasonic technology by an ultrasonic reactor working as anti-scaling technology in pipelines, especially those with high carbonate content.

II. MATERIALS AND METHODS

A. *Materials*

1) *Calcium carbonate scaling plant:* An inorganic salt scaling plant, as shown in figure 1, was developed to study the phenomenon of carbonate deposits in pipes and heat exchangers by reacting calcium chloride bi-hydrate ($CaCl_2 \cdot 2H_2O$) and sodium bicarbonate ($NaHCO_3$). The system has three basic operation modes: (i) scaling, (ii) cleaning and (iii) flushing configured by a set of manual valves.

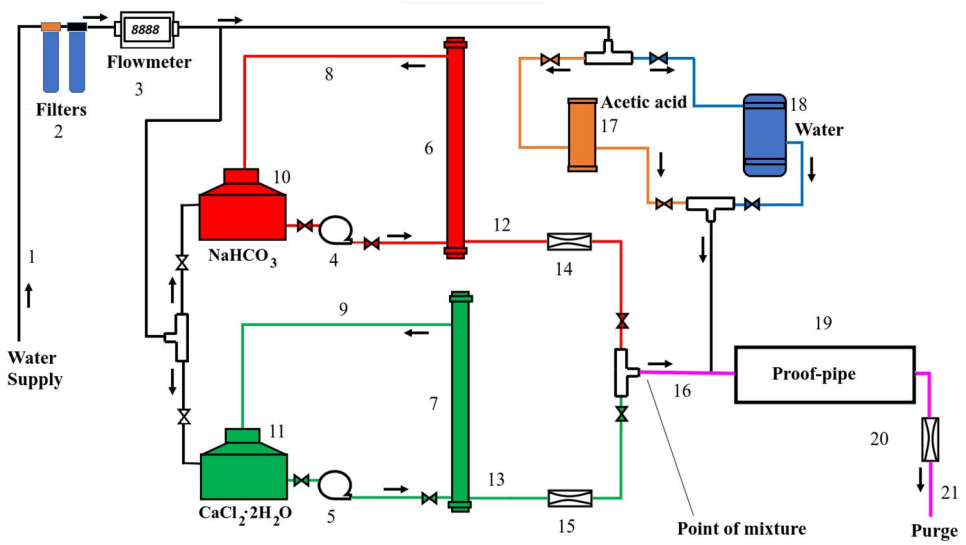

Fig. 1 Flowchart of the calcium carbonate scaling plant.

The reservoir of each reagent was fed with drinking water (1) properly filtered (2). The amount of water on each tank was determined by measuring the filling flow rate, using a flowmeter model K24 manufactured by Shengze® (3). The system was designed to ensure equal flow rate for each reagent. Recirculating pumps (4, 5) carry each reagent through lift columns (6, 7). The level of the reagents in these columns is kept constant by overflowing pipes (8, 9), returning reagent excess to storage tanks (10, 11). Reagent levels in the columns were kept equal and stable. Thus, the flow rates of each reagent, defined by gravity flow conditions, will be equal and stable as well. The outlet pipes of each column (12, 13), made with equal length, had their flow rate measured by the flowmeters (14, 15), and were connected to each other at the mixing point (16).

For the cleaning and subsequent water flushing of the proof-pipe, two other reservoirs were used. One containing acetic acid (17) solution, used as calcium carbonate solvent, and the other containing water (18). Both solvent and water circulate by gravity through the proof-pipe.

The reaction products were discharged (21) after circulating through the proof-pipe (19). A flowmeter (20) was used to measure the total flow rate, confirming the sum of the flow rates of each reagent. The calcium carbonate scaling plant is shown in figure 2.

Fig. 2 Image of the calcium carbonate scaling plant.

2) *Ultrasonic Reactor:* The developed ultrasonic reactor consists of three high power ultrasonic transducers Langevin type, power of 60 W each and resonance frequency of 40 kHz, with a maximum operating temperature of 65 ºC, manufactured by Yongda Ultrasonic®. The reactor central cavity was made in 6063-T5 aluminium (A) where the fluid pass through from (B) to (I). Transducers (C) are fixed by screws to an AISI-304 stainless steel plate (thickness of 1.5 mm) (D). Aluminium frames (E) are used to distribute bolt stress over the sealing gaskets (F). An air purge outlet (G) has been provided to ensure exclusively presence of liquid inside the cavity. Finally, a drain outlet (H) allows remove calcium carbonate crystals excess (see figure 3). The device was designed with reduced dimensions for preliminary tests on the scaling plant described above, allowing study on antiscaling technologies at 1 MPa maximum operating pressure. An ultrasonic generator model ARS-QXDY (3000 W), adjustable on frequency from 20 to 45 kHz, manufactured by OURS ULTRASONIC®, was used for power supply. The generator was tuned to supply 90 W to the transducers.

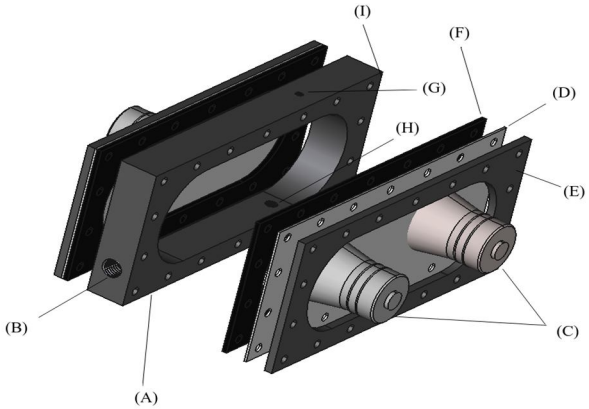

Fig. 3 Ultrasonic reactor: (A) central body, (B) fluid inlet, (C) transducers, (D) stainless steel plate, (E) frame, (F) rubber gasket, (G) air purge, (H) drain, (I) fluid outlet.

As transducer characteristics can vary according to physical coupling, each transducer was calibrated after mounting by adjusting its torque, to operate close to its original characteristics (40 kHz x 20 Ω). A TRZ analyzer, manufactured by Physical Engineering®, was used to measure the impedance and resonance frequency. Table 1 resume results found after this adjustment for each transducer.

TABLE I
Resonance Frequency and Impedance for Each Ultrasonic Transducer Adjusted After Mounting.

Transducer	Resonance frequency (kHz)	Impedance (Ω)
1	39,5	80
2 (central position)	39,0	110
3	39,4	85

3) *Proof-pipe:* A transparent polyvinyl chloride pipe (PVC) was assembled as proof-pipe allowing the calcium carbonate crystals visualization during the precipitation process, see Figure 4. The translucent PVC pipe has 6.40 mm internal diameter, 1.50 mm thick wall and 5 meters long, this flexible pipe was rolled in spiral shape. The pipe was attached to a plastic plate and supported vertically. Threaded connections were installed at its ends enabling interconnection to the scaling plant.

Fig. 4 Spiral type proof-pipe used in the tests.

4) *Data Acquisition System:* The data acquisition system is composed of an ESP-32 microcontroller model WROOM-32 responsible for acquiring the flow rates of each reagent (sodium bicarbonate and calcium chloride) as well as the total flow rate of the system. The signal from each sensor was conditioned through a 5.0 to 3.3 Vdc logic converter to match microcontroller input, see Figure 5. A specialist supervisory system was developed for data storage and monitoring on Matlab® using the Design App tool.

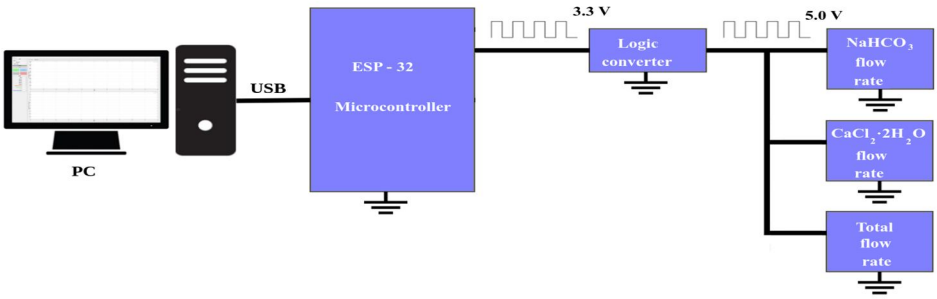

Fig. 5 Flow rate data acquisition system.

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5) *Reagents:* The preparation of the reagent solutions starts with making a saturated solution of calcium chloride dehydrated and sodium bicarbonate, which were made by diluting pure and analytical salts of $CaCl_2 \cdot 2H_2O$ and $NaHCO_3$ in deionized water at 25 °C until precipitation appears at the bottom of the beaker. The saturated solutions of calcium chloride and sodium bicarbonate were filtered through a 1 µm mesh paper filter to remove any precipitation and other potential contaminants. These saturated solutions were then diluted again on drinking water until the final concentration was reached: 12.6 g/L for the $NaHCO_3$ and 7.35 g/L for the $CaCl_2 \cdot 2H_2O$. Table 2 shows the reagents' properties. Final diluted solutions may rest for at least three days to reach complete single-phase stability and full reacting power.

TABLE II
Reagent Properties.

Property	$CaCl_2 \cdot 2H_2O$	$NaHCO_3$
Concentration (g/L)	2.24 ± 0.08	9.15 ± 0.10
Molarity	0.06	0.15
Conductivity (µS/cm)	9.50 ± 0.35	10.87 ± 0.39
pH	7.20 ± 0.20	8.70 ± 0.20
Temperature	23.0 ± 0.5	

In order to determine solutions' reactivity and stability as a function of preparation resting time, a method based on light transmission and a test equipment (both patent pending) were developed. These tests were conducted by mixing equal volumetric portions of diluted reagents and monitoring its light transmission pattern for 1800 seconds. This procedure was repeated for solution resting time varying from 0 h up to 144 h, as shown in figure 6. In this graph, the digital results were artificially displaced to facilitate their evolution visualization as a function of time for different reagents resting times. It is possible to observe that the curve behavior changes as a function of time and there is almost no change for 48 h on. After this period, the solutions showed regularity in their crystal production and precipitation.

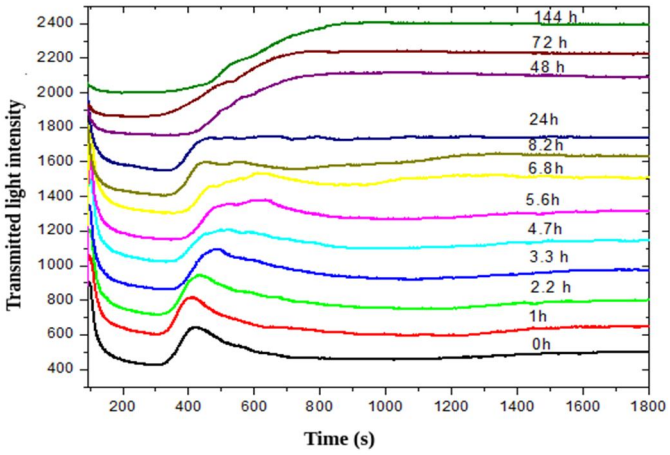


Fig. 6 Optical evaluation of reagents reactivity.

B. Methods

1) *Sound field assessment tests:* The internal sound pressure field zones able to produce cavitation were evaluated by erosion of 0.5 mm aluminium foil positioned inside the ultrasonic reactor through the gravimetric method. Two acrylic frames were used to support the aluminium foils in its right position and granted the tests repeatability, see figure 7. Aluminium foils were weighed on a semi-analytical balance (0.0001 g resolution), model Q510L210C manufactured by Quimis®, before and after the tests, once dehydrated.

Fig. 7 Plastic frames for erosion tests on aluminium foil.

2) *Scaling tests:* Tests were carried out with the scaling plant to compare calcium carbonate deposition on the proof-pipe surface at two conditions: with and without US irradiation. The flow rates of each reagent were monitored by turbine type flowmeters, model YF-S402 manufactured by Sea®, inside measuring range of 0.2 to 6.0 L/min (accuracy of ± 3%), to ensure reagents equal flux. Reagents total flow rate (sum of the flow rate of each reactant) allowed the monitoring of the pressure drop on the test-pipe. Initially, the system worked without any scaling prevention, afterwards ultrasonic reactor was connected to the system, 200 mm before the proof-pipe inlet, see figure 8. Temperature on the transducers surface was measured in real time by a FLIR thermographic camera, model i5 (resolution 0.1 ºC).

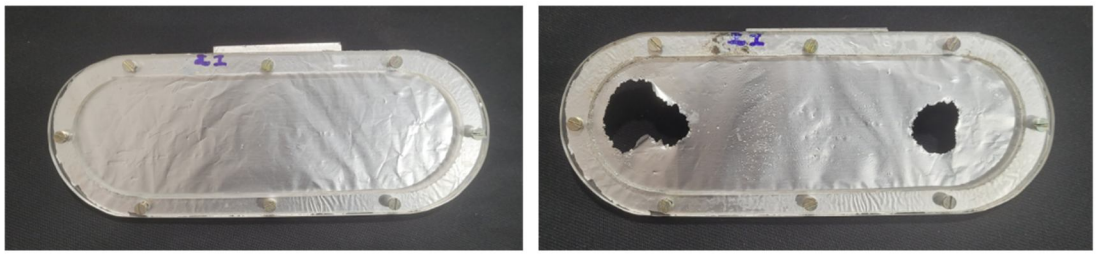
Fig. 8 Ultrasonic reactor assembled in the calcium carbonate scaling plant.

The tests stop conditions were defined as maximum experimental time of 180 minutes or flow rate reduction of 50% of its initial condition, which indicates partial pipe blockage. Prior to each test, the reagents mixing point, the proof-pipe and the purge were properly cleaned with diluted acetic acid (5%) for 15 min and 30 min of water flushing. At the end of each test, the produced fluid was collected, filtered under vacuum on a membrane filter (mash of 0.47 µm) and examined by scanning electron microscope (SEM) to assess crystal morphology. A JEOL microscope model JSM-6610LV, equipped with an energy dispersive spectroscopy (EDS) module, from Oxford Instruments, was used.

III. RESULTS

A. *Sound pressure field analysis*

Figure 9 shows the erosion on aluminium foil after 5 minutes of sonication inside the ultrasonic reactor in stationary regime. Intense cavitation is observed at regions close to the three transducers, especially regarding the two external transducers. At the central region (transducer 2), the tape was not drilled. The high impedance obtained during the resonance frequency calibration process (see Table 1) may have contributed to the low performance of this transducer.



(a) (b)

Fig. 9 Erosion tests on aluminium foil: a) At initial condition; b) After 5 min of sonication.

Mass removed by ultrasound cavitation during the experiment is shown in figure 10. After 4 minutes, the erosion process tends to saturation. Probably, after 4 minutes, there is no available metallic material in the ultrasonic cavitation. After 5 minutes the relative mass reduction was 6.4 %.

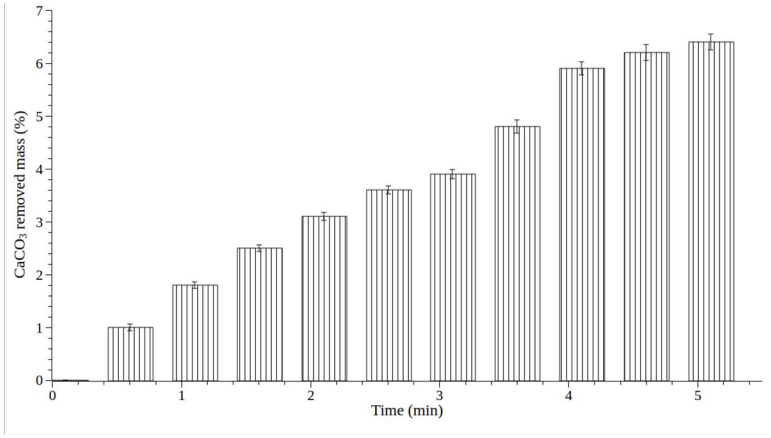


Fig. 10 Aluminium foil mass removed over time.

B. *Tests of the Ultrasonic Reactor as Antiscaling Device*

As commented on the chapter B (heading 2), the surface temperature of the transducers was measured in real time by the rmographic and did not exceed 39 ± 3 ºC during 3 h of testing, as shown in figure 11.

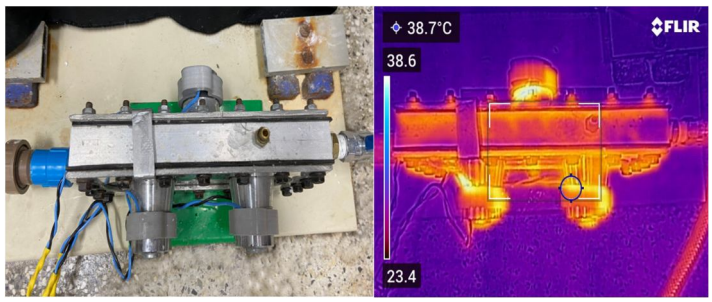


Fig. 11 Measurements of the transducer surface temperature.

Figure 12 shows the proof-pipe after continuous deposition tests without ultrasound.

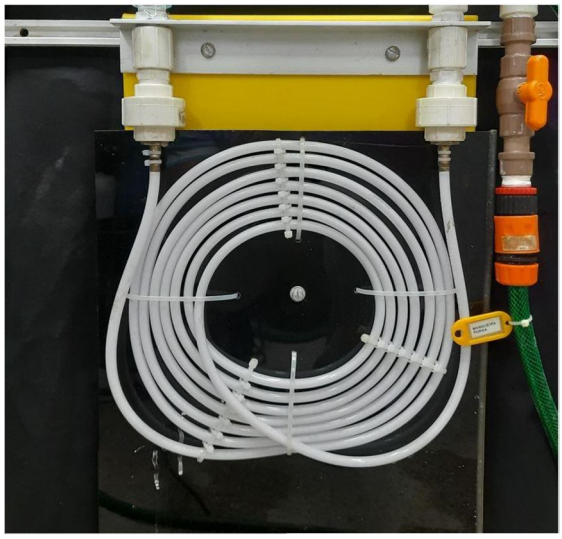


Fig. 12 Sample pipe after scaling tests without US.

The flow rate as a function of time for both tests is shown in figure 13. The maximum flow rate, without any scaling prevention device (1.86 L/min) is reached quickly, however, a continuous and well-marked reduction in the flow rate is verified, imposing the end of the test after 140 min, when the flow rate was reduced to 0.94 L/min. Once the ultrasonic reactor was mounted on the hydraulic system, the flow rate shows a certain inertia and grows up to 1.76 L/min. The use of ultrasound increases carbonate crystals precipitation that probably increased pipe internal friction, and therefore, imposes pressure drop in the beginning of the test. In addition, the ultrasonic reactor increased minor hydraulic losses, justifying the reduction in maximum flow rate. However, one can see an oscillatory behavior of the flow rate when ultrasound was applied (1.56 - 1.76 L/min). US generates crystals with less adherence to the internal surface of the pipe and, consequently, this newly produced and weakly adhered material can be dragged as soon as pressure drop increases momentarily. The pressure rise upstream of the flow contributes to dragging particulate material restoring original flow. At 140 minutes, during sonicated test, the flow rate was 1.62 L/min or 8% of relative reduction. The ultrasonic reactor allowed a reduction of approximately 42% in hydraulic resistance if compared to the condition without scaling prevention. The collapse of the bubbles generated by cavitation induces shock waves that contribute to temperature increasing, decreasing molecular force and surface tension. The adhesive force is reduced, so deposited scale will be separated from solid surface as a result of the vibration induced by the ultrasound, decreasing the possibility of carbonate crystal deposition on the proof-pipe internal surface [16]. Da Silva *et al.* [21] found similar results, obtaining lower pressure drop and scaling rate of calcium carbonate crystals in pipes when testing the effectiveness of an ultrasonic reactor in a carbonation line.

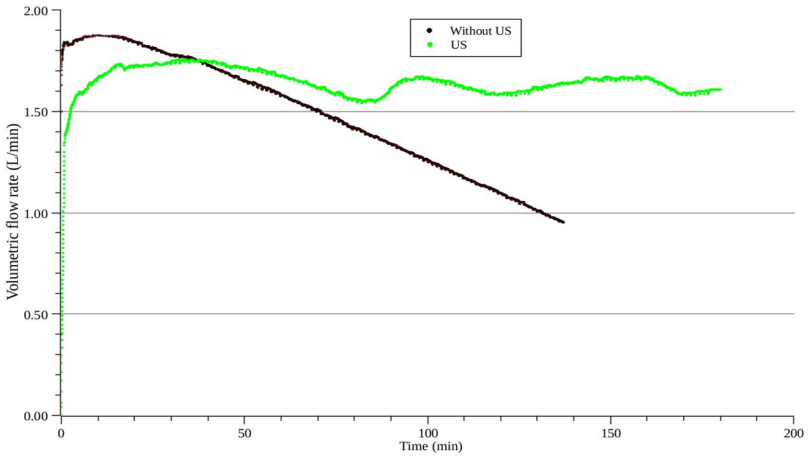


Fig. 13 Total volumetric flow rate of the system over the time.

Figure 14 shows details of the particulate material adhered to the proof-pipe with and without ultrasonic irradiation. A very fine-grained particulate material and sparsely distributed over the pipe inner surface can be verified, justifying the flow rate behavior when ultrasound was applied.

The SEM images are shown in Figure 15, there is a well-marked reduction in particle size. An average value of 7.6 µm was verified when the test was carried out without any descaling device, whereas a value of 1.8 µm was found when ultrasound was applied, which means 76% reduction in particle size. In addition, a significant rounding of the produced particles was observed under ultrasonic irradiation, which may reduce adhesion to proof-pipe surface. Other authors found similar results [22] - [24].

Aiming long-term production equipment protection, the anti-scaling developed device can be associated with an inertial separator, which can drain ultrasound induced crystals, allowing it to be purged before avoiding dragging along the pipe, when the system operates over a long period.

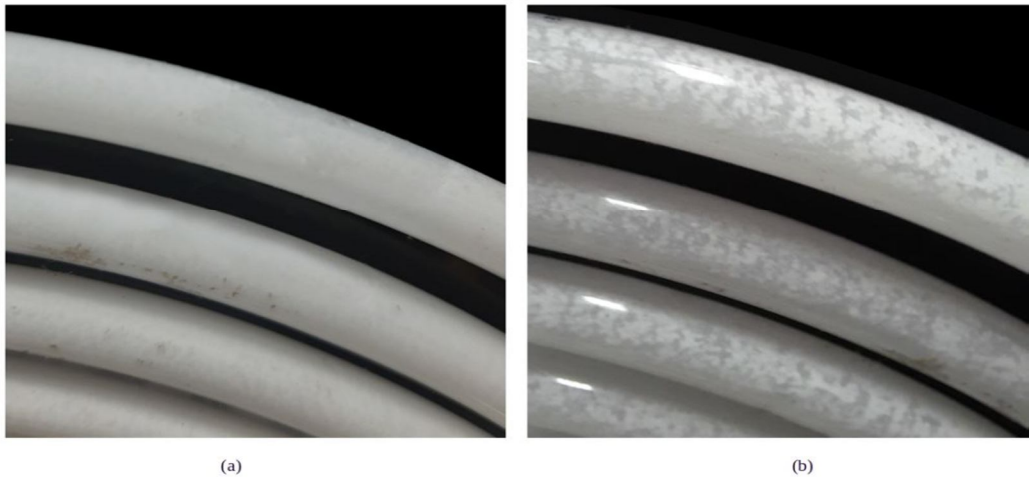


Fig. 14 Details of the particulate material adhered on the proof-pipe: (a) without US; (b) under US.

When ultrasound is transferred to the liquid, some effects such as mechanical vibration, heating and cavitation, can occur and in consequence influencing crystallization of ions Ca^{2+} and HCO^{3-} present in solution[5]. In addition, the shock waves generated by cavitation increase the solution kinetic energy, increasing insertion of solute molecules on all crystal faces, creating of a more regular structure[25]. Other authors justify this rounding on particle shape as a consequence of particles fusion by collision, induced by ultrasound pressure field [26].

As mentioned by Lawrence and Sydney [27], calcium carbonate crystals generated without ultrasound (figure 10-a) show a rhombohedral topology, usually the calcite phase. When ultrasonic radiation is applied during calcium carbonate crystals formation, the predominant structure is orthorhombic, typical of vaterite phase [28].

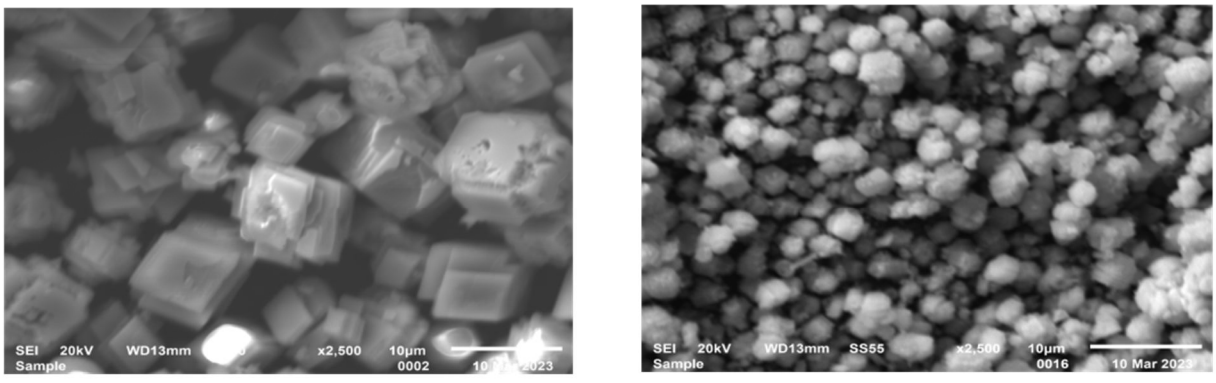


Fig. 15 SEM images at 2500x: a) Without US. b) With US.

IV. CONCLUSIONS

Calcium carbonate scaling is a serious problem for industrial installations. Mechanical techniques to control and remove this deposition are important tools to avoid equipment and pipeline failures.

This work evaluated the performance of an ultrasonic reactor as calcium carbonate descaling device using flexible plastic pipe as a proof-pipe in a hydraulic system specially designed to create scaling in pipes and heat exchangers.

The system worked under two different conditions. Initially, the system was tested without any scaling prevention device. After that, a reactor was mounted endowed of three Langevin 60 W @ 40kHz, powered by an ultrasonic generator at 90 W x 40 kHz. Sound field pressure tests on aluminium foil, with pure water, indicated strong cavitation activity inside the reactor. Mass reduction of 6.4% was observed after 5 minutes sonication.

The US reactor was tested hooked up to the calcium carbonate scaling plant to determine its performance as a descaling device at a condition like that found in oil extraction industries. No transducers overheating was verified. The flow rate monitoring during reaction between sodium bicarbonate and calcium chloride solutions showed that the reactor was able to maintain stable total flow rate, even after 3 h of testing. No line blockage was verified during the test. After 140 minutes of test, without sonication, already presented a reduction of 50% in flow rate, leading to the stop of the test, while, at that same elapsed time, in ultrasound presence, only a reduction of 8% in the total flow rate has been observed. Hydraulic resistance reduction of 42% was verified under US irradiation. US generates cavitation and induces shock waves that decrease molecular force and surface tension of fluid, reducing adhesive force of crystals, contributing to the deposited incrustations separation from the solid surfaces and reducing the possibility of carbonate crystals deposition on the pipe surface.

SEM analysis showed drastic reduction (76%) in particle size and significant rounding of the crystals under US action. The modifications on crystal shape verified point to changes in crystal nucleation process, particles fusion by collision caused by cavitation, in addition to higher mixing between the solute molecules and formed particles.

Results show ultrasound as an interesting alternative for scaling prevention, minimizing obstruction by calcium carbonate in pipelines and increasing availability of flow equipment in industrial facilities.

V. ACKNOWLEDGEMENT

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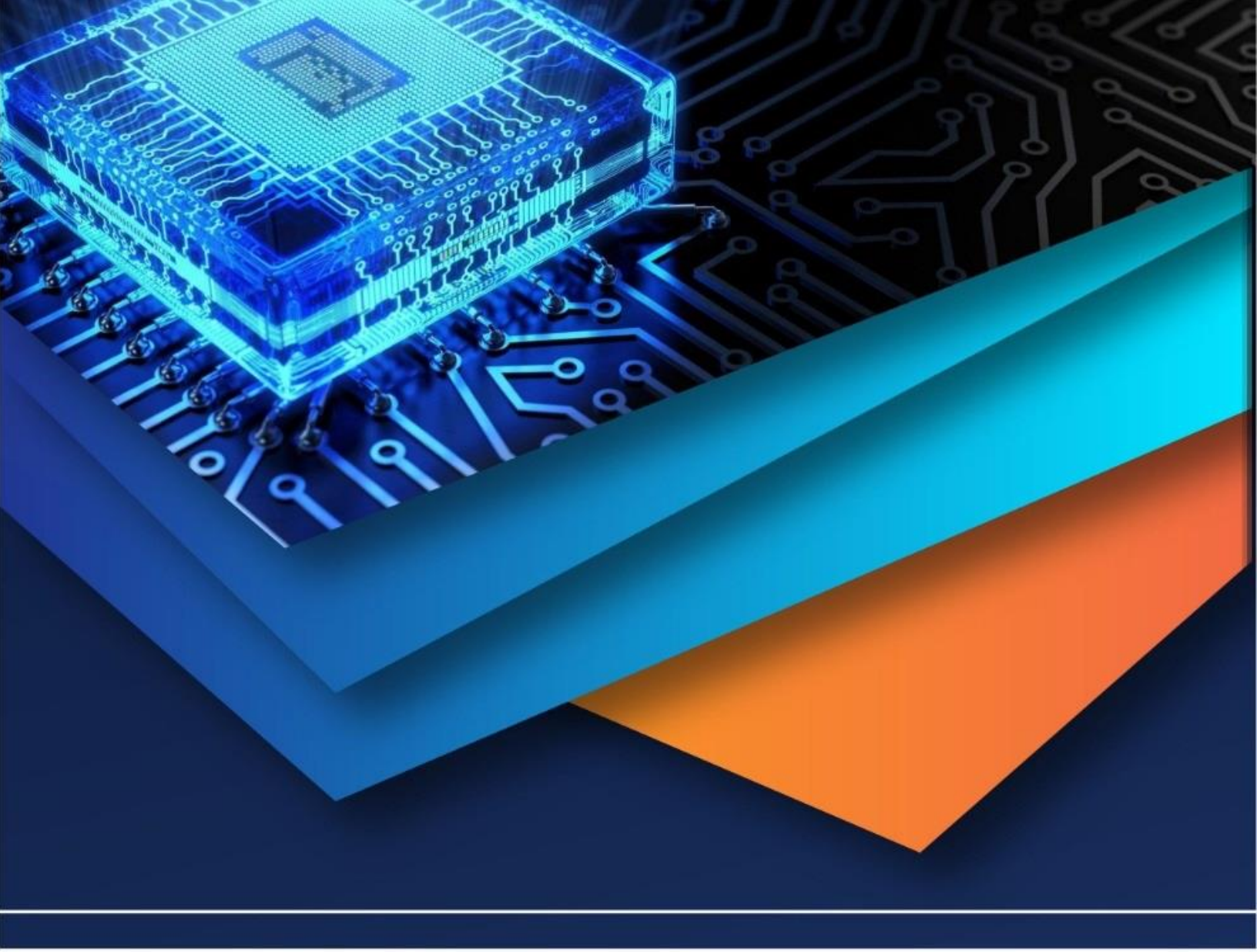

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