Development of an experimental methodology for assessing the growth of scale (CaCO₃) in pipelines

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Abstract. One of the problems that affect the mechanical integrity of pipelines transporting aqueous fluids is the formation of fouling deposits as $CaCO_3$. Today different theoretical models that establish criteria trend but are not decisive in establishing the true rate of growth of scale in a pipeline there. This paper presents experimental development which was conducted to deposit scale of calcium carbonate by using a loop in which the deposits formed into a capillary of stainless steel AISI 316 and thus study the influence of various physical - chemical factors such as the composition of the solution, flow rate, and surface temperature and the presence of CO_2 generated on amount of scale. The evolution of the thickness and the amount of fouling obtained by tests was subsequently determined and by X-ray diffraction (XRD) and scanning electron microscopy (SEM) were characterized the deposits. With the results of the study show a strong influence of carbon dioxide in the amount of fouling obtained and according to the composition of the solution the flow rate fouling can generate greater or lesser scale. Under the test conditions is not possible to obtain deposits at room temperature.

1. Introduction

Pipes that transporting aqueous fluids are affected their mechanical integrity due to the formation of scales, which occur when the fluid is oversaturated by compounds having in solution and this starts at the time when variations occur in the operational conditions of system (pressure, temperature, etc.) [1]. The literature indicate that the operating conditions have an important effect on the growth of the scales in the industries [2].

Deposits occur in different types of industries in which a solution aqueous is in contact with a surface [3], said layers may be formed in pumps, pipes, heaters, heat exchangers, boilers, evaporators and various accessories and equipment [4]. Among the industries most affected are those using equipment heat transfer and extraction and production of oil [5], causing costs due to production losses, removal treatments (chemical or mechanical) and repair or replacement of equipment. In industrial processes can present different types of fouling as strontium sulphate, barium sulphate, calcium sulphate and calcium carbonate [6], the latter is one of the most common in the petroleum industry.

Due to problems caused by scales, posits have gained a lot of research have get deposits under laboratory conditions in order to understand the causes, variables and trend criteria involved in the formation, growth and removal of the layer formed. Methods of studying the deposition of layers in the laboratory are: electrochemical techniques, rotating ring electrode, micro quartz crystal balance (QCM) [7] and loops, these last time-consuming for the test performance [8], but considered as a subject of study time of blockage of the pipes can establish good predictions. Zhang et al. [1], showed the performance of a kinetic model to predict the rate of fouling of calcium carbonate, which is based

on measurements of growth of single crystals of calcite, the results for the model were compared to those obtained with experiments and obtained a good correlation between the blocking time predicted by the model and obtained from laboratory tests also evaluated the potential application of the model to compared the results with the profile of scale that had an oilfield.

In this paper we present experimental development used to study the formation of calcium carbonate scale on steel AISI 316 with a loop and correlated different physicochemical factors such as the composition of the solution, flow rate, temperature of the surface and presence of CO_2 on the amount generated of scale.

2. Experimental equipment and procedure

The substrate on which were deposited of the scales was a capillary of 15cm length and 4.1cm internal diameter, made of stainless steel AISI 316. The test tube was attached to a pump to impulse the solution to inside of capillary and back to the vessel which initially contained. To increase the temperature of capillary was used a heating bath, a schematic representation is shown in Figure 1.
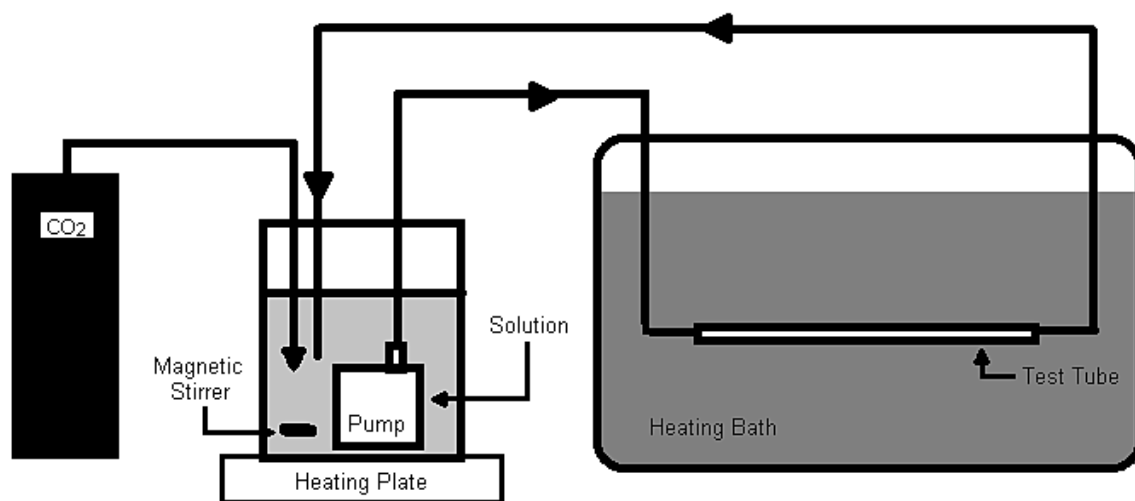

Figure 1. Experimental assembly for the precipitation of calcium.

In the performing test two different solutions were used: first consisting of H_2O-$NaCl$-$CaCO_3$ and second: $CaCl_2.2H_2O$-$MgCl_2.6H_2O$-$NaHCO_3$-$NaCl$-H_2O [9]. For solutions analytical grade reagents which were weighed on a sartorius balance AC121S with a precision of four tenths were used.

The experiments were conducted for 101 hours, during this time the heating bath temperature was controlled by an immersion thermometer, test at room temperature (18-21), 60 and 80$°C$ and velocities of 0.0063 and 0.035m/s which cause laminar flow which creates favourable conditions for scaling are executed. Besides was injected bubbling controlled of CO_2 a container containing the fouling solution when required it. After each test performed the evolution of scale thickness obtained was measured, taking data every centimetre of capillary length, this was done by means of a microscope stereographic SMZ800 NIKON. For the characterization of the deposits obtained X-ray diffraction was used by a team PANalytical X'Pert PRO with copper anode and a sweep between 5 and 90° driving a step time of 0.5 seconds.

3. Results

The evolution of scale obtained reveals growth as presented in Figure 2 and Figure 3 shows measurements of the layer thickness.

The results obtained through this study are presented in Table 1 for each correlation variables used, the obtained maximum thickness in micrometres and the amount of fouling micrograms is shown.
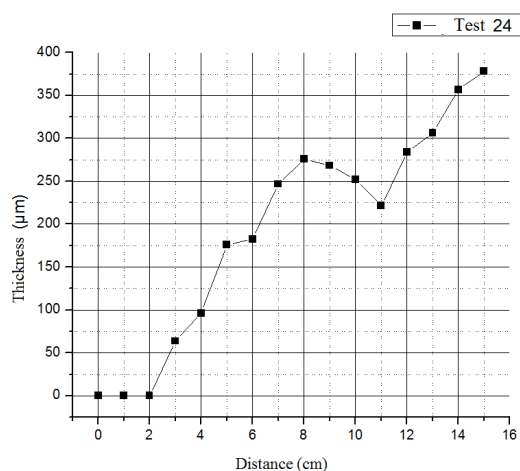
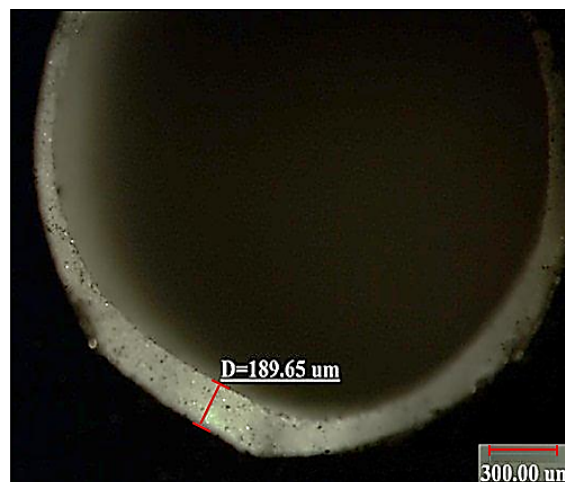**Figure 2.** Thickness vs distance for the test capillary 24.

Figure 3. Thickness 14 centimetre length of capillary for test 17.

Table 1. Maximum thickness and amount of scale for each of the tests.

P.No.	Conditions				Thickness	Amount
	T SP [a]	VF (m/s) [b]	CS [c]	CO_2	(μm)	(μg)
9	60°C	0.0063	1	Yes	193,04	1896,03
10	60°C	0.0063	1	No	nm [d]	nm [d]
11	60°C	0.0063	2	Yes	426,20	2707,98
12	60°C	0.0063	2	No	256,12	2217,53
13	60°C	0.035	1	Yes	31,24	117,06
14	60°C	0.035	1	No	nm [d]	nm [d]
15	60°C	0.035	2	Yes	329,39	2818,99
16	60°C	0.035	2	No	147,08	1331,16
17	80°C	0.0063	1	Yes	267,37	2152,23
18	80°C	0.0063	1	No	nm [d]	nm [d]
19	80°C	0.0063	2	Yes	111,24	1024,08
20	80°C	0.0063	2	No	173,70	1345,36
21	80°C	0.035	1	Yes	146,70	1028,79
22	80°C	0.035	1	No	nm [d]	nm [d]
23	80°C	0.035	2	Yes	222,28	2011,53
24	80°C	0.035	2	No	377,92	2918,92

For test between 1 to 8 is not get scale measurable.
a Temperature of the surface
b Flow rate
c Composition of the solution
d Scale obtained unmeasurable

SEM photograph in the Figure 4 indicated that the deposit structure in uniform and that in few places is possible observed crystals of $CaCO_3$ completely build. Too by XRD, it was determined that for tests with solution 1 calcite precipitated it in Figure 5 is observed and solution 2 identified compounds as: calcium carbonate as aragonite, double carbonate of calcium and magnesium, magnesium carbonate as magnesite and calcium. We conclude that aragonite precipitated because that the presence of Mg^{+2} inhibits the growth of the calcite [10], and dolomite and magnesite exists by partial or complete replacement of calcium by magnesium in the calcium carbonate.
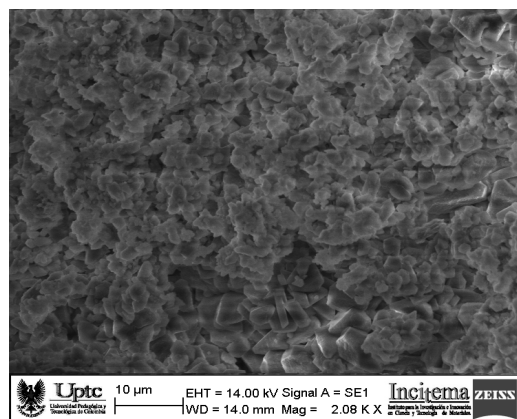
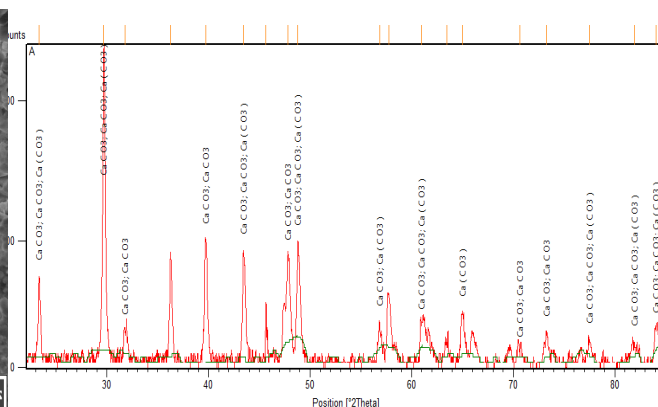**Figure 4.** Scanning electron microscopy picture of CaCO$_3$ crystals.

Figure 5. X-ray diffraction (XRD) for the scale of CaCO$_3$.

4. Conclusions

Due to the different nature of the solutions different thicknesses with solution 1 and 2 are presented and also as fluids were not filtered, the flow rate generates different cutting forces due to the particle size of each solution because differences grades solubility of the reagents used in each.

Solution 1 generates no measurable deposits without the presence of CO$_2$ this occurs because the low solubility their reagents, which is enhanced when carbon dioxide is present.

The higher the temperature the energy of the system increases causing higher speed chemical reactions. It also generates a decrease of solubility causing increased degree of supersaturation, so it requires use temperature above room ambient so that the deposits are formed.

With solution 2 is obtained aragonite which its crystallographic system is orthorhombic generates a deposit more porous that obtained with solution 1 because this is entirely formed by calcite.

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