ASSESSMENT OF SCALE FORMATION IN THE COLUMN OF AN OIL AND NATURAL GAS PRODUCING WELL: A CASE STUDY

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ABSTRACT

The present study investigated the influence of depth, pressure and temperature on the composition and behavior of scale formation along a column in a well that produces oil and natural gas. The objective was to improve treatments to remove precipitates formed on the column surface. Ten samples of residues were collected from several points along the production column (from 15.4 m to 4061.5 m) that exhibited scale formation or corrosion problems. Dissolution tests were performed in organic acids. The predominant composition was defined and confirmed by dissolution assays and characterization was determined using X-Ray Diffractometry (XRD), X-Ray Fluorescence Spectrometry (XRF) and Scanning Electron Microscopy (SEM). Residues with distinct characteristics were observed in different proportions, showing a tendency to increase or decrease in mass with depth. The results indicate that correlations can be established between the type of scale formation and depth and, consequently, thermodynamic conditions (pressure and temperature).

KEYWORDS

scale formation; production column; XRD; XRF; SEM

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1. INTRODUCTION

Corrosion, inorganic and organic deposits and formation of stable emulsions often compromise oil flow, or even plug columns, lines and underwater and surface equipments (Oliveira, 1997).

A decline in production may be caused by damage or elevation problems within the formation (Mackay et al., 1999). Inorganic salt deposits, commonly known as scale, are some of the possible problems encountered during exploration and production, and can lead to reduced permeability in the hydrocarbon-producing formation due to the precipitation of inorganic salts in the reservoir, and perforations in the rock, production column and some surface equipments (Dyer and Graham, 2002; Marques et al., 2001; Mackay et al., 2003; Jordan et al., 2000; 2004).

Formation waters associated with hydrocarbons normally contain large concentrations of chloride (Cl^-) and sodium (Na^+) ions. Also present in significant concentrations are sulfate (SO_4^{2-}), bicarbonate (HCO_3^-), carbonate (CO_3^{2-}), calcium (Ca^{2+}), magnesium (Mg^{2+}), barium (Ba^{2+}), potassium (K^+), strontium (Sr^{2+}) and bromide (Br^-) ions. The most common scale formations found in oil fields contain calcium carbonate (CaCO_3), calcium sulfate (CaSO_4) and barium sulfate (BaSO_4) (Chen et al., 2005; Eidelwein, 1992; Jordan et al., 2004; López-Sandoval et al., 2007; Salman et al., 2007). The solubility of these substances may be influenced by factors such as pressure (P) and temperature (T). The total mass precipitation depends on the degree of saturation (D.S.) of the medium and the disturbance to which the system is subjected (variations in turbulence, pressure, flow velocity, presence of nucleating agents, etc.). As the degree of saturation increases, the solution becomes supersaturated and, depending on P and T conditions, may lead to precipitation of excess salt. This is one of the primary causes for the occurrence of inorganic deposits (Akin and Lagerwerff, 1965; Chen et al., 2005; Mucci and Morse, 1983).

Calcium carbonate formation is a function of the combination of calcium ions (Ca^{2+}) and bicarbonate ions (HCO_3^-) present in water, or indirectly, by the action of carbon dioxide (CO_2) from formation, which interact reversibly as shown by Equation 1

(Akin and Lagerwerff, 1965; Chen et al., 2005; Mucci and Morse, 1983; Tomson et al., 2003; Zhang et al., 2001):

Other types of rarely seen scale formation such as zinc sulfates, lead sulfates and nickel sulfates, usually associated with carbonates, have increased interest among researchers (Collins and Jordan, 2003; Jordan et al., 2000; Jordan et al., 2004). Thus, scale formation and inhibition mechanisms have been extensively investigated for several decades (Eidelwein, 1992; Jordan et al., 2000; Jordan et al., 2004; Tomson et al., 2003), in an attempt to address the numerous resulting problems which can have an impact on operational costs due to lost production, interventions in the wells, the need to remove chemicals, etc. The most widely used prevention method is the chemical treatment with inhibitors of scale formation (Salman et al., 2007). There is ample evidence in the literature (Chen et al., 2005; Collins and Jordan, 2003; López-Sandoval et al., 2007) that low concentrations of metal ions, including Zn^{2+} , Cu^{2+} , Fe^{2+} , Fe^{3+} and Mg^{2+} , may affect nucleation and the CaCO_3 precipitation rate, inducing morphological alterations. Some scale formations are easily removed by acidification. Calcite, however, is difficult to predict using the existing models. The best way to identify the presence of carbonate is through monitoring.

The proper characterization of scale formation, which is very important in determining salt deposition on the internal walls of a well, is a serious operational problem for the oil industry due to lost productivity and time spent on equipment cleaning. This issue has been extensively studied in recent years, since oil companies are obtaining increasingly complex products and tend to produce more and more residues.

With the purpose of assessing the development of scale formation and showing the importance of characterization in a production column, we sought to associate the influence of depth, pressure and temperature on the composition and phases of scale formation, and suggest the most adequate treatment for their removal.

2. MATERIALS AND METHODS

2.1 Extraction of the organic phase

The procedure for extraction of the organic phase was carried out with the soxhlet method (Avanti Soxtec 2055). To do this, 5.0-g samples were extracted from each pre-determined depth with chloroform, which is the least toxic solvent among others tested and provides a more efficient extraction (Moura et al., 2006). The cycle of extraction was optimized for 120 minutes at a temperature of 140°C. Then the sample was cooled and pulverized in a ball mill, model RETSCH PM 200 planetary, at 500rpm for 10 minutes to obtain a lower average grain size (100 # mesh).

2.2 X-ray Fluorescence Spectrometry (XRF)

X-ray fluorescence spectrometry was conducted using a Shimadzu XRF-1800 spectrometer in the semi-quantitative mode, in which the wavelength dispersion methodology (WD-XRF) was employed. The detection limit of the device ranges from sodium ($\text{Na} \rightarrow Z = 11$) to uranium ($\text{U} \rightarrow Z = 92$), using $\text{RhK}\alpha$ radiation ($\lambda = 0.615\text{\AA}$). The sample was pressed with boric acid, using a 6:1 ratio (boric acid:sample), with two 60-second pressing cycles and a load of 30kPa.

2.3 X-ray Diffraction (XRD)

X-ray diffraction was performed using a Shimadzu LabX XRD 6000 diffractometer, with

$\text{CuK}\alpha$ radiation ($\lambda = 1.5418\text{\AA}$). The diffractogram obtained was analyzed by Search Match 3.01 and Shimadzu Basic Process software with the JCPDS database (Joint Committee on Powder Diffraction Standards). Thus, qualitative information on scale formation and/or corrosion products can be available for the various crystalline phases present.

2.4 Scanning Electronic Microscopy (SEM)

Scanning electronic microscopy was carried out using a Philips XL-30 ESEM in low vacuum mode (environmental mode), which does not require sample metallization. Fragments of each sample were extracted (Soxhlet), removing organic matter to avoid contamination of the equipment. In this analysis, samples were not pulverized, allowing the study of scale formation, morphology and chemical composition using EDS (Energy Dispersive Spectrometry).

3. RESULTS AND DISCUSSION

3.1 Operational conditions of the production column

Fig. 1 illustrates the dynamic gradient profile of temperature-depth, showing that temperature increases with depth along the column, in accordance with the geothermal gradient of the oil field. According to Graham and Williams (2005), the primary elements responsible for scale

Figure 1. Temperature behavior along the column (Bolonhini, 2008).

Figure 2. Pressure behavior along the column (Bolonhini, 2008).

formation are abrupt changes in pressure and temperature during production, mainly due to alterations in the carbonate system. As the fluid flows through the production column, it passes through zones of lower temperature and pressure, releasing CO_2 from the water produced along with the gas and condensate.

Fig. 2 shows a practically linear variation of the pressure gradient in the entire fluid column in the well. These data are presented simulating the layout of the well, with the y-axis depicting depth and the x-axis depicting pressure. It can be seen that pressure increased linearly with depth until reaching the production zone. Villela (2004) reports that it would be normal for the dynamic pressure gradient not to be linear, since there is a progressive release of gas and consequent reduction in column fluid density. However, the graph in Fig. 2 is linear, suggesting that there is only the predominance of monophasic flow in the column. According to Silva (1993), it is common practice to interpret pressure tests in gas and condensate reservoirs considering monophasic gas flow.

The formation of calcium carbonate scale in production columns is related to gradual depressurization, which occurs along the column toward the surface. The solubility of CO_2 and CaCO_3 is strongly dependent on pressure and temperature (Eidelwein, 1992). Depressurization along the column from the bottom of the well

removes CO_2 from the solution. This shifts equilibrium in the direction of calcium carbonate formation, increasing pH and CaCO_3 precipitation, according to Equation 1.

3.2 Characterization of residues

During removal of scale formation residues from the production column, 78 samples were collected from the internal walls, at points between 15.4m and 4061.5m. Because of the large number of samples, they were separated into groups with the same visual aspects according to the depth range. In this case, 10 samples with different aspects were selected to represent the groups; however, all samples were individually characterized.

The characterization of residues aims to determine how they are disposed along the production column. The following techniques were used: XRD, to identify the phases present; XRF, to determine the elements present; and SEM, to study the morphology of the residues.

Table 1 shows representative samples of each depth range along the production column, except sample A10, which was collected near the perforation after removal of the column.

Fig. 3 shows the macroscopic characteristics of samples classified in each group. It can be observed that with an increase in depth, the residues on the

Table 1. Samples selected and their depth ranges.

Sample	depth range (m)
A1	15.40 – 55.90
A2	65.40 – 108.65
A3	322.05 – 766.80
A4	1617.45 – 1792.05
A5	1811.45 – 1908.45
A6	1918.15 – 1994.00
A7	2003.70 – 2100.70
A8	2110.40 – 2236.50
A9	2255.90 – 2535.95
A10	3987.00

production column display different aspects. Fig. 3(A1) depicts samples collected near the surface, exhibiting similar characteristics (large thin plates). Fig. 3(A2) presents iron-like characteristics (small thin orange plate-like encrustations). Fig. 3(A3) samples resemble sample 3(A2), but are more crumbly, their surface is rougher and they exhibit lighter color at the point of contact with the column. Samples from Figures 3(A4), (A5) and (A6) display similar characteristics (plate-like and granules, distinguished by roughness and color).

Fig. 3(A7) shows a different aspect from the others, with typically lighter granules in some of the plates. These are clear, well-defined crystals. The depth range where most of the restriction occurred was between 1850m and 2120m, where

scale formation was irregularly-shaped. At this range, the thickest scale formation was found at 2062m. Samples from Figures 3(A8), 3(A9) and 3(A10) exhibited very different characteristics from those of the previous samples, with darker and smaller plates.

3.3 Solubility assays

Solubility assays in acid were conducted, similarly to those carried out in the removal of scale formation [acetic acid 5% (v/v) + 7% formic acid (v/v)]. The images in Fig. 4 show the difference in reactivity between the groups near the head and at the bottom of the well. However, dissolution reactions of samples A1, A2, A3, A4, A5, A6 and A7 are quite severe, due to the reaction of the acid-carbonate mixture in the scale formation with the consequent release of CO₂. However, samples A8, A9, A10 did not react with the acids.

3.4 XRF studies

The results of X-ray fluorescence (XRF) are presented in relative mass percentage. This means that the analysis must be made by comparing the relationship between percentage mass values of each element for every sample. The XRF results obtained in this study were not normalized to 100%, deducting the values of elements with low molecular weight (which are not detected by the equipment).

Again, the XRF values are presented simulating the layout of the well. The y-axis depicts depth, where point 0 (zero) is the head of the well and

Figure 3. Visual aspects of samples collected along the column (the scale shown in red is in centimeters).

Figure 4. Behavior of the samples during the acidification reaction with organic acids.

4061.5m is the bottom of the well; the x-axis shows the percentage of each element analyzed in all the residues collected. Thus, it is important to determine how the elements in the scale formation are located in the production column.

Fig. 5 shows the percentage of lead (Pb) at different depths along the column. Despite exhibiting low concentrations, a number of oscillations can be observed, but from sample A7 onward its percentage tends to increase gradually, except the last sample (A10) at 3986 m. Sample A9

shows the higher percentage of Pb (2.33%) at a temperature of 160.96°C and pressure of 80.89kgf/cm².

Fig. 6 depicts the rise in molybdenum (Mo) percentage with an increase in depth, except the last sample (A10), at 3986m. The presence of Mo was surprising, since it is not normally found, and because the percentage (2.80%) was close to that of the alloy material used in the column ([Santanna et al., 2003](#)); in sample A9 the percentage was 2.29%. Since sample A10 was not collected directly

Figure 5. Variation of lead percentage with depth.

Figure 6. Variation of molybdenum percentage with depth.

Figure 7. Variation of nickel percentage with depth.

from the column wall, the Mo percentage decreased to 1.13%.

Fig. 7 shows that nickel (Ni) behaves similarly to Mo. But in sample A10, at a temperature of 180.66°C and pressure of 114.92kgf/cm², the percentage of Ni increased to 6.15 %. The presence of nickel in the scale formation residue may originate by the dissolution of the metal alloy in the production column (Santanna et al., 2003).

Because of the acidification with HCl (15%), which aimed to clean the deepest regions of the well, and given that the column was in contact with the strong acid solution for an extended period of time, it is believed that chromium (Fig. 8), in addition to Mo, Pb and Ni, originated from dissolutions of the metal alloy in the column (Santanna et al., 2003), since similar percentages of these elements were detected in the alloy. It is therefore surmised that the column was oxidized at depths around 2500m.

Figure 8. Variation of chromium percentage with depth.

Figure 9. Variation of zinc percentage with depth.

Figures 8 and 9 show high percentages of chromium (Cr) and zinc (Zn), observed at depths of more than 2000m, that is, from sample A7 onwards there is continuous increase in both Cr and Zn contents, reaching 28% and 32%, respectively. The high Cr percentage may be associated with oxidation in the production column, which underwent acid attack (HCl, 15% v/v) in the deepest region. It is suggested that these results be compared with physical analyses of samples from the column itself. The high percentage of Zn

detected in the scale formation at a depth around 2500m is likely attributed to low ZnS solubility at high temperatures and pressures.

Fig. 10 shows the behavior of calcium (Ca), one of the most commonly found elements in the column (about 45%), declining to approximately 20% below a depth of 2400m (sample A9), when the column temperature reaches 160.96°C and the pressure is 80.89kgf/cm². This high percentage of Ca explains the evolution of gaseous CO₂ in the

Figure 10. Variation of calcium percentage with depth.

Figure 11. Variation of iron percentage with depth.

solubility tests of organic acids (Figure 4), due to the calcium carbonate present in the samples.

The results of the iron (Fe) content analysis in the scales (Fig. 11), shows that iron is the predominant element in the sample (A10), which is probably the main cause of the corrosion on the metal surface.

Finally, Fig. 12 illustrates the variation of the sulfur (S) percentage along the column. This behavior is similar to that of zinc and iron (Figs. 9

and 11), that is, its percentage increases with depth, reaching approximately 13%. The presence of sulfur, zinc and iron is associated with the high pressure and high temperature in the well.

3.5 XRD studies

The diffractogram obtained for sample A1 is shown in Fig. 13, and the pattern corresponds to a predominantly crystalline material consisting primarily of calcium carbonate (calcite). The XRD results corroborate the XRF data (Fig. 10), which

Figure 12. Variation of sulfur percentage with depth.

Figure 13. Diffractogram of sample A1 representing group I (the largest phase).

indicate that the percentage of calcium was 46.13%.

In Fig. 14, the diffractograms show that new peaks, corresponding to new phases, emerge for samples collected from deeper points. Moreover, from sample A6 onward low-intensity peaks of iron sulfide (FeS) and zinc sulfide (ZnS) appear, and suggest the existence of trace compounds. From sample A7 onward, that is, at depths below 2000m, the intensity of peaks related to these compounds

starts to increase in the diffractograms, and the galena (PbS) peak emerges as trace compounds, since its intensity is very low, whereas the intensity of the calcite peak (CaCO_3) decreased.

The most common forms of iron, pyrite (FeS_2) and iron oxide, were not found. However, the literature (Jordan et al., 2000; 2004) reports that zinc sulfide contains up to 36% iron. Since the crystals were quite characteristic, it is believed that the iron in the spectra is zinc sulfide.

Figure 14. Diffractograms of selected samples.

Figure 15. Scanning electron microscopy micrographs of samples (a) A1 and (b) A9.

Samples A8, A9 and A10 contain different proportions of ZnS, showing a tendency to increased mass with depth. In addition to ZnS, there is a small amount of PbS, which may indicate that zinc comes from the formation. However, further data are needed to provide a more thorough conclusion, such as the presence of zinc in formation water and knowledge on the geochemistry of the reservoir rock.

3.6 SEM studies

Scanning Electron Microscopy (SEM) and Energy Dispersion Spectroscopy (EDS) images were used to obtain information on morphology and elementary semi-quantitative composition of solid residues attached to the inner walls of the well.

The SEM data generally corroborate those obtained with the XRD and XRF techniques.

Figure 16. Micrographs of sample A9 with details (a) of galena crystals (PbS) at 2000x amplification, and (b) zinc sulfide crystals, at 1000x amplification.

Figure 17. Micrograph of both sides of sample A9 at 20x amplification.

However, sample A1, near the surface, exhibited homogeneous structures (Fig. 15) and the predominant matrix is calcium. Sample A9 shows the presence of crystals in a Ca matrix.

Fig. 16 shows details of sample A9, with well-defined galena (PbS) crystals, a cubic structure almost always in octahedra and zinc sulfide (ZnS) crystals.

Fig. 17 shows the images obtained on both sides of sample A9. On side 1, incrustation is on the tube

wall (visually smoother surface), while on side 2 it is located inside the column (visually rougher surface).

Details observed on side 1 reveal a homogeneous surface, suggesting a predominant element. The spectrum shows that the primary element is chromium, possibly due to scale formation in the production column, which is a metal alloy containing more than 20% chromium (Santanna et al., 2003).

Figure 18. Amplification of side 1 (250x) of sample A9, with EDS spectra highlighted.

Fig. 18 presents the micrograph of side 2, scale formation in contact with the fluid, showing the presence of more than one element, due to the different densities in the elements. The spectra of the highlighted regions primarily show the presence of calcium, considered as the matrix, as well as sulfur and lead, with the same energy levels.

- Homogeneous surfaces with calcium carbonate crystals (CaCO_3) were observed by SEM in samples at depths with lower pressure and temperature (A1 to A6). Subsequent samples primarily exhibited ZnS and PbS crystals.
- The presence of a high percentage of chromium in sample A9, in contact with the metal alloy of the column, may be associated to its degradation.

4. CONCLUSIONS

Based on the results of this study, the following conclusions can be drawn:

- Samples of scale formation at lower pressures and temperatures, from A1 to A6, contain a higher percentage of CaCO_3 .
- The presence of calcite (CaCO_3) was confirmed by dissolution tests in a 5% (v/v) solution of acetic acid and 7% (v/v) formic acid and by XRD and XRF tests. However, samples A8, A9 and A10 exhibited low dissolution in the same solutions.
- The presence of ZnS, PbS and FeS was detected in samples of scale formation at greater depths, pressure and temperature (A7, A8, A9 and A10).
- X-ray fluorescence spectroscopy analyses revealed higher levels of zinc, chromium and sulfur in scale formation samples A8 and A9.

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