

Combined Application of Inhibitors for Scale and Corrosion Mitigation: Lessons Learned

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ABSTRACT

Scale formation in surface and subsurface installations of geothermal plants diminish economic efficiency by reducing operational performance and create following-up costs related to maintenance, cleaning and disposal. The application of efficient antiscalants and inhibitors creates additional costs but can be efficiently balanced by the overall benefits of efficiency improvement and reduction of follow-up costs.

Currently, antiscalant and inhibitor tests are conducted at three of the geothermal plants of the Upper Rhine Graben (URG) within the framework of the German funded project SUBITO (FZK 0325790). The products are tested either solely or in combination with a second product by using separated injection points.

All tested products and product combinations showed different results. Three of the antiscalants reduced the total mass of deposited scales efficiently but were partwise incompatible with the thermal brine at high temperatures. In consequence, deposits with high Total Organic Carbon (TOC) were deposited upstream, blocked filters and build up deposits at the entrance of the tubular heat exchangers.

After careful pre-selection, corrosion inhibitors were applied at all three geothermal plants in combination with antiscalants for barium sulfate inhibition. As a result, the total mass of deposits was significantly reduced which was one of the major goals of the extensive product tests. Moreover, the successful utilization of corrosion inhibitors for metal-scale inhibition proves the direct link between corrosion processes and scale formation. It highlights therefore the importance to understand the complex interactions in a geochemical system on both levels chemical reactions and physical parameters.

1. INTRODUCTION

1.1 Consequences of Scaling and Corrosion in Geothermal Power and Heat Plants

Economic feasibility of a geothermal power plant relies on continuous and constant operation of the geothermal loop including surface and subsurface installations. Constraints of operational performance like unscheduled shut-down periods, intense maintenance, cleaning operations and follow-up costs reduce their reliability and result in significant loss of revenues. Disposal of scales which contain heavy elements and even radionuclides is cost and time intensive and requires high administrative work load. The same applies to equipment which is polluted with this type of scales.

Scaling and corrosion affect geothermal sites in different ways. Scaling, in most cases, reduces operational performance over time and requires intense cleaning procedures, either mechanical or chemical.

Corrosion, especially localized corrosion like pitting, crevice or galvanic corrosion leads to unscheduled shut-down times due to leakages or as prevention in order to avoid leakages. Localized corrosion occurs even at low uniform corrosion rates and is more difficult to treat chemically than uniform corrosion.

Scaling and corrosion phenomena are often interrelated processes. On one side, scaling can trigger localized under-deposit corrosion and has the potential to create small scale galvanic cells in case of heavy metal deposition. On the other side, corrosion processes change geochemical conditions like Eh and pH at the metal/brine boundary at the micro level which can trigger scale formation.

1.2 Geothermal Power and Heat Plants of the Upper Rhine Graben

The Upper Rhine Graben (URG) is a geological structure in western Europe, extending nearly 300 km N-S with a maximum opening of 40 km. It was formed by a Tertiary rift system and is connected to a

geothermal anomaly with higher heat flow than in surrounding areas resulting from fluid convection in naturally existing fracture system.

Today, five geothermal plants are operating in the Upper Rhine Graben (URG), three on the German side and two, including one plant for solely heat production, on the French side, Figure 1. Several new projects are planned in this area and two of them are already close to realization.

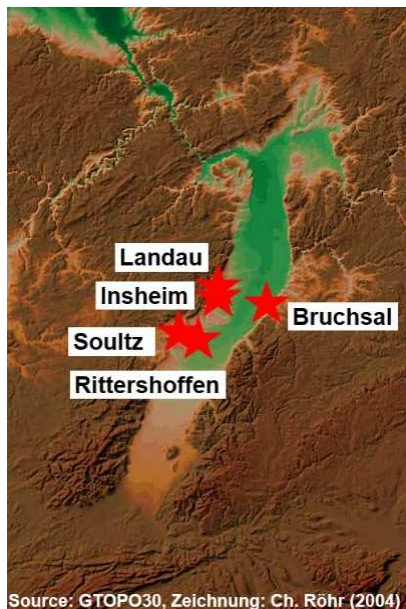


Figure 1: Geothermal Power and Heat Plants in the Upper Rhine Graben, (modified after Röhr, 2004).

1.3 Scaling and Corrosion in Geothermal Plants of the Upper Rhine Graben

Typical scales in geothermal plants of the URG consist mainly of barium/strontium sulfates (barite/celestine) and minor amounts of deposits, rich in metals which contain lead sulfide, arsenic and antimony, Figure 2.

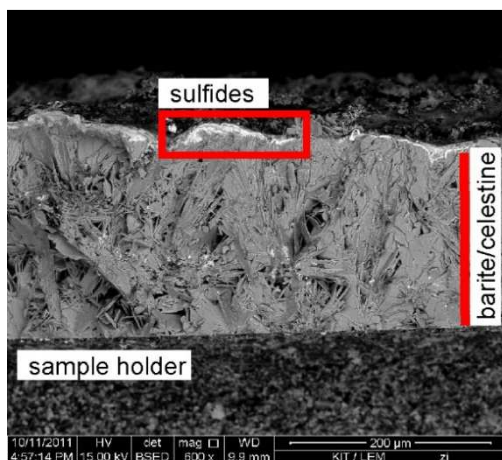


Figure 2: Scanning Electron Microscopy exposure of a barium sulfate scale cross-section with minor amounts of metal-rich deposits in the back scattered mode (BSED), (after Scheiber, 2012).

Successful mitigation of barium sulfate was reached by using antiscalants like phosphonic acid at dose rates below 10 ppm, (Scheiber et al. 2015). Metal rich scales which contained a large amount of lead sulfide (PbS) became the dominant deposit in the geothermal sites of the URG.

Both scale types, sulfates and sulfides, are formed due to the temperature decrease in the heat exchangers by exceeding their saturation. Therefore, heat exchangers, and all surface and subsurface installations of return line and injection well are affected by scale deposition. Uniform corrosion of mild steel surface piping in geothermal plants of the URG is known to be very low (0.15 mm/y) but pitting and crevice corrosion occurs at low temperatures of the return line, (Battici et al. 2010 and Mundhenk et al. 2013), Figure 3.

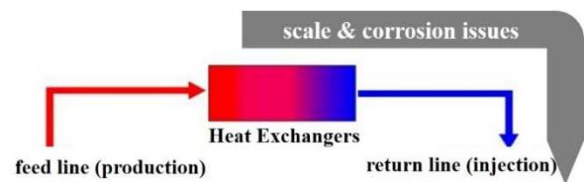


Figure 3: Corrosion and scale related issues occur mainly in heat exchangers, return line and injection well.

In the URG scale forming processes of Pb, As, Sb rich deposits were not fully understood in the past. The presence of lead sulfide indicated also the presence of S^{2-}_{aq} in the brine which was never detected. Moreover, H_2S was never detected to be a part of the Non-Condensable Gases (NCG) of the brine.

The Na-Ca-Cl brine is highly mineralized, up to 105 g/L at the western rim of the URG and 125 g/L at the eastern rim (Scheiber et al. 2015, Sanjuan et al. 2016, Jähnichen et al. 2019). The brine contains 1.0-1.3 norm liter (NL) NCG per 1 L brine (~0.17 mass percent) and consists with more than 95% of CO_2 . In comparison to geothermal high enthalpie fields which contain gas loads of up to 2.5-4.0 mass percent the gas load of the URG seems low but in combination with high calcium concentration of up to 8000 mg/L calcite scales are formed very fast in the production well and feed line if brine is not properly pressurized.

All geothermal plants of the western rim of the URG use line shaft pumps (LSP) for brine production to increase production flow rate but also to mitigate calcium carbonate precipitation by pressurizing the brine above the Gas Breakout (GBO) pressure which varies as a function of the gas load. Typical GBO pressures at the western rim of the URG are 18-20 bars which means that brine is produced at pressures of 20-22 bars.

Pressurizing the brine above the GBO does not only mitigate calcium carbonate formation, it also decreases CO_2 induced corrosion significantly. Operating the geothermal plants close or even below the GBO results in an increase of localized corrosion phenomena at the return line downstream of the heat exchangers.

1.4 Antiscalant and Inhibitor Testing Program: SUBITO – Sulfide Inhibition and Disposal

Barium sulfate inhibition in geothermal power and heat plants of the URG is already applied as state-of-the-art method. Efficiency of the treatment is a barium sulfate mitigation > 98%.

Exotic scales like lead sulfide are very difficult to mitigate by standard chemical measures like antiscalants or dispersing agents and are therefore not included in the standard portfolio of service companies for water treatment in geothermal and in oil and gas. The URG is a challenging environment for chemical brine treatment due to

- production temperatures of up to 170°C.
- high salinity of > 100 g/L Total Dissolved Solids (TDS).
- calcium concentration of up to 8000 mg/L.
- complex scale mineralogy.
 - barium sulfate
 - lead sulfide
 - Pb, As, Sb accumulation but (in 2014) unidentified redox state and unknown mineralogical composition of deposits
 - minor amounts of Fe, Cu and Si but unknown associated minerals
 - associated deposition of Pb-210 and Ra-226
- Germany – Palatina region: restrictions of water authorities. Water treatment in geothermal is restricted to products with WGK 1 (Water Hazardous Classification).

The research project *Sulfide Inhibition and Disposal* (SUBITO (FKZ 0325790A)) was created out of the difficulty to establish efficient brine treatment in the URG which tackles both mineral types, sulfates and sulfides. At that time, it was assumed, based on elemental analysis, that As and Sb also form sulfide deposits.

The aim of the research project is to reduce total scale mass and to eliminate or at least to limit the accumulation of toxic and radioactive elements by using products with specific abilities like antiscaling, filming and dispersing properties.

One of the most crucial tasks for the project was the requirement to keep up the continuous successful barium sulfate inhibition at any time during product tests. As a side effect of barium sulfate formation, the radionuclide Ra-226 gets trapped in the crystal lattice by chemical substitution of barium and the sulfate scales have to be considered as low-level radioactive waste (Scheiber et al., 2012).

During the first phase of the project antiscalants which were selected specifically for sulfide mineral mitigation were tested followed by the combined application of antiscalants with corrosion inhibitors in the second phase:

- Phase 1 – antiscaling / dispersion.
- Phase 2 -antiscalants + corrosion inhibitors.

Product tests were performed at two geothermal sites of the URG, the geothermal power plants of Insheim (Germany) and Soultz-sous-Forêts (France).

2. PRE-STUDIES: INHIBITOR SELECTION AND LABORATORY TESTS

2.1 Inhibitor Selection

Product selection and dosage adjustment is a question of plant location and its geochemical conditions but if brine chemistry does not vary too much, specific product groups can be applied in a larger geological region.

In total seven service companies for water treatment were contacted or contacted project partners themselves for product recommendations. Out of the seven six companies recommended one or several products for testing either solely scale inhibition or solely corrosion inhibition or both.

2.1 Laboratory and Compatibility Tests

Laboratory pre-tests covered the following tasks:

- brine / inhibitor compatibility – calcium tolerance at temperature conditions of the return line (injection conditions).

High calcium concentration at high temperatures are extremely challenging conditions for antiscalants and corrosion inhibitors. Especially at the dosing point high inhibitor concentrations are present which do not correlate to the intended dose rate at full dilution.

- effect of the product on barium sulfate inhibition

Antiscalants and scale inhibitors which use the dispersing effect can reduce product efficiency by interaction. Especially when using corrosion inhibitors, the efficiency of the antiscalants for barium sulfate mitigation must be confirmed.

- in case the recommended product had the ability for barium sulfate inhibition:
 - efficiency for barium sulfate inhibition in comparison to the already applied and successful product

As already stated, barium sulfate inhibition in the URG is associated with the presence of Ra-226. Due to the fact that barium sulfate mitigation has been already established with an efficiency of more than 98% the application of new or additional products are not allowed to limit this efficiency in any way.

Exemplarily the laboratory pre-test for barium sulfate inhibition efficiency (IE) is described for the combined tests of antiscalants and corrosion inhibitors. The aim of the investigations was to review the effect of

different corrosion inhibitors on the already applied barium sulfate inhibitor (reference inhibitor). Therefore, three solutions were prepared and mixed during the experiment:

- artificial brine
- barium chloride solution
- sodium sulfate solution

The artificial Na-Ca-Sr-Ba-Cl-SO₄ brine was prepared with supersaturation of BaSO₄, which correspond in a simplified way to the brine chemistry of the Insheim geothermal power plant. In order to simplify the ion diversity in the original solution, the K⁺ concentration was converted into Na⁺ concentration. Correspondingly, all bivalent ions except for strontium were converted into Ca²⁺ concentration. Strontium correlates with the original fluid concentration. The adjustment of the model solution made sure that its ionic strength matches the one of the original brine, Table 1. NaCl (Geyer p.a.), CaCl₂·2H₂O (Geyer p.a.) and SrCl₂·6H₂O (ACS reagent grade, Merck) were applied. For this specific experiment the barium and sulfate was enhanced to four times of the original concentration in order to speed up the chemical reactions.

Table 1: Composition of the artificial Na-Ca-Sr-Cl solution (pH=5.2 @ T=20.0°C) in comparison to the original brine composition from Insheim (main components only).

Variables	units	Artificial Brine	Original Brine
Na ⁺	mg/L	33 325	30 800
K ⁺	mg/L	-	4 200
Ca ²⁺	mg/L	8 850	8 770
Mg ²⁺	mg/L	-	115
Sr ²⁺	mg/L	438	438
Ba ²⁺	mg/L	140	35
Σ Cations		42 613	44 358
Cl ⁻	mg/L	67 307	65 300
SO ₄ ²⁻	mg/L	620	155
HCO ₃ ⁻	mg/L		156
Σ Anions		67 307	65 611
TDS	g/L	110	110
I	mol/L	2.13	2.13

Inhibitor combinations of laboratory pre-tests:

- Reference: barium sulfate inhibitor
- Comb 1: corrosion Inhibitor 1a + reference
- Comb 2: corrosion Inhibitor 3 + reference
- Comb 3: corrosion Inhibitor 4 + reference

Since the active concentration of the commercial products were not known for all products, the inhibitor concentrations (IC) IC = 10 mg/L were used for all products in the experiments.

For evaluation, the results of the reference inhibitor were compared with the results of the product combinations.

After heating 50 ml of artificial Na-Ca-Sr-Cl solution to 60 °C in a tightly closed glass tube, the inhibitors were added, followed by the Na₂SO₄-solution and the BaCl₂- solution. The moment after brief mixing of all different solutions was declared as reaction start t = 0.

The samples were left in an incubator (Certomat H from Fa. Braun) at a temperature of T=60°C±0,5°C under dynamic conditions (n = 145 rpm, Certomat S) for about 48 h. Turbidity measurements, conducted with a photometer (SQ 118; Merck), were used to determine the reaction progress of the barium sulfate formation.

In dynamic bottle tests the first hours after dosing (t ≤ 6 h) are crucial for the inhibitor application and efficiency. Therefore, sampling was carried out hourly after reaction start and the experiment was finished after 45 h. After sampling the solution was immediately filtered (0.45 µm) and diluted to 1:100 with 0.75% HNO₃. Residual concentration of barium and strontium was determined with ICP-MS.

The reaction progress of the thermodynamically supersaturated barium sulfate solutions was monitored by turbidity measurements. With the creation of more and more solid matter extinction increases. Figure 4 shows the extinction as a function of reaction time of the different inhibitor combination in comparison to the Reference. During the first four hours no visible solids were formed in all solutions which was confirmed by extinction measurements. After this time the extinction increased for Comb 2, Comb 3 and Reference. After a reaction time of 8 hours, more and more large crystals were formed and some of them got even attached to the tube wall. Therefore, turbidity does no longer correlate to the actual precipitation of barium sulfate. For Comb 1, the extinction value is nearly constant <0.5 during the total reaction time of 48 h, no turbidity was observed at all.

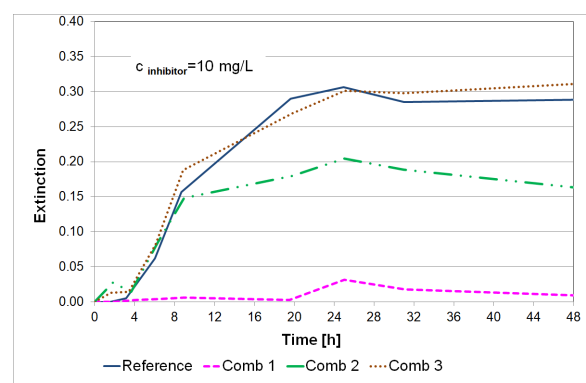


Figure 4: Extinction as a function of the reaction time. initial Ba²⁺ c = 140 mg/l at T = 60°C (±0.5°C) and a shaking velocity of 145 rpm.

Respectively six of the samples have been analysed for the remaining Ba²⁺ and Sr²⁺ concentration in solution. Figure 5 shows the inhibitor efficiency (IE) of the

tested inhibitor combinations and the barium sulfate antiscalant as a function of the remaining Ba^{2+} concentration. The higher the concentration the better IE.

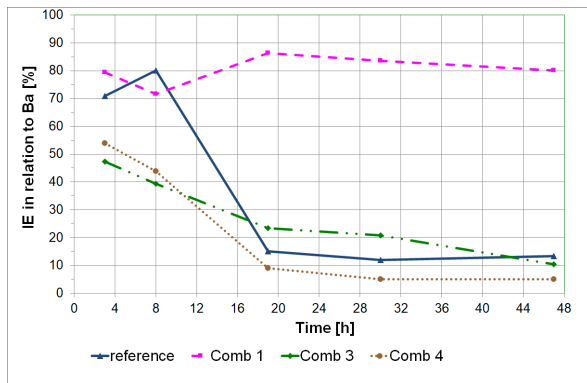


Figure 5: Inhibitor efficiency (IE) in relation to the analyzed residual Ba^{2+} concentration in the solutions.

Results of the turbidity measurements are confirmed for Comb 2. Efficiency of the reference inhibitor was reduced by the corrosion inhibitor of about 50% during the first eight hours which was also observed for Comb 3. This is difference to the extinctions curves of Comb 3 but can be explain by formation of smaller and therefore more crystals due to the presence of the corrosion inhibitor.

The slope of Comb 1 shows that barium sulfate precipitation can even be efficiently retarded for a very long-time period even in the presence of very high supersaturation of fourfold the original concentration of Ba^{2+} and SO_4^{2-} .

The reaction progress of this experiment showed clearly the influence of corrosion inhibitors on the efficiency of the barium sulfate antiscalant. Two of the product combinations reduced efficiency to about 50% during the crucial first 8 h. This means that power plant operators must be prepared to possibly increase dose rate of the barium sulfate antiscalant when applying corrosion inhibitors of Comb 2 and Comb 3. Comb 1 on the other hand seems to support and stabilize the efficiency of the barium sulfate inhibitor which was confirmed by both turbidity and concentration measurements.

This type of laboratory experiment is a powerful tool for a pre-selection of appropriate product candidates for the field test.

Out of logistical and time related reasons only Comb 2 was tested at Insheim and the corrosion inhibitor of Comb 3 was tested in combination with another barium sulfate antiscalant at Soultz which is also described by Mouchot et al. 2019.

3. ON-SITE TESTS OF ANTISCALANTS AND CORROSION INHIBITORS

3.1 On-site Test: Selected Products

Out of more than twenty product, antiscalants and corrosion inhibitors, eleven were tested on-site either solely or in combination:

- “sulfide” antiscalants: 7x

Scale inhibition of barium sulfate and lead sulfide combined in one product.

Inhibitor 1, Inhibitor 2, Inhibitor 4, Inhibitor 8, Inhibitor 10, Inhibitor 11 and Inhibitor 12.

- corrosion inhibitors: 4x

Additive 3, Additive 4, Additive 5, Additive 7.

Barium sulfate mitigation is achieved successfully with the injection of phosphonic acid (Scheiber et al. 2015). Two different products were applied at Insheim and Soultz to whom the description Reference 1 and Reference 2 refers to.

3.2 Brine/Inhibitor Compatibility

Three of the tested products were highly incompatible with brine of the URG at high temperature (feed line – production side). Two of the products were applied at Soultz (Inhibitor 10 and Inhibitor 11) and the other one at Insheim (Inhibitor 1). Inhibitor 1 was tested successfully at Soultz and proofed its ability for significant mass reduction of the scale deposits. Therefore, it was also recommended for the test at Insheim. Production temperature of Insheim is higher than the one at Soultz, with a difference of 16 K. At Soultz no incompatibility was observed but at Insheim the Δp of the production filters increased within the first ten days of injection and the test of this product had to be aborted immediately due to severe incompatibility issues. The inhibitor clogged the complete production filter with a brittle and sticky deposit and contained 23 mass% of TOC, Figure 6.



Figure 6: Incompatibility of brine/antiscalants at high temperatures and high calcium concentration at the geothermal power plant Insheim.

This type of deposit can be assumed also to be present in all surface installations upstream to the heat

exchanger and even inside the heat exchanger to the point where the brine cools down below 150°C which corresponds to the Soultz production temperature at which it was fully compatible with the brine.

The distance between inhibitor injection point and the filters is approximately 3 m, which is considered to be sufficient for brine / inhibitor mixing. The formation of the deposits indicates a strong incompatibility of at least one or even more components of Inhibitor 1 at temperatures of 163°C.

The incompatibility of three different products from three different service companies showed the urgency for compatibility tests at production temperature before field application even if the retention time at this temperature is below 50 s as it is the case for Insheim.

3.3 Scale Monitoring and Mass Balance

Scale monitoring of the different product tests was realized by sampling scales from a pipe of the surface installations. Specifically for the research project SUBITO the power plant operator of Insheim and Soultz agreed to install bypass systems which allows to operate the monitoring pipes in the framework of the normal surface installations which had the same flow, temperature and pressure conditions as the connected equipment, Figure 7.

Each product was tested for several weeks to several months. Before starting the product injection either a new pipe was installed or the pipe was cleaned rigorously by water jetting.



Figure 7: Pipes of the return line for scale monitoring at the geothermal site of Insheim.

One parameter of efficiency evaluation was the mass balance of scale formation as a function of the deposited mass per 150 cm² surface area per accumulated brine volume during the time of exposure and dose rate of the products, Figure 8.

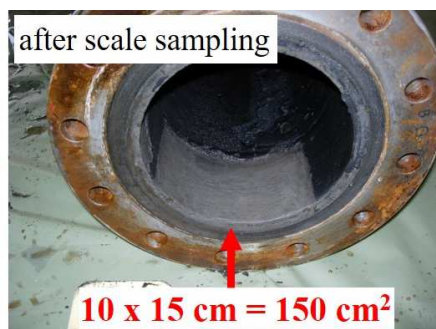


Figure 8: Monitoring pipe of the geothermal power plant of Insheim after scale sampling.

Sampling was realized after pipe deinstallation. The attachment of the scale to the surface, the scale morphology, its color and thickness was monitored every time. The results of the Soultz “pipe-monitoring” are part wise summarized in Mouchot et al., 2019.

At Insheim, the injection of the reference inhibitor (solely barium sulfate antiscalant based on phosphonic acid) lasted for twenty-nine months during the project duration of SUBITO and was considered as long-term monitoring. Within the first 4 months dark grey and soft scales were deposited with a total thickness of nearly 4 mm, Figure 9. The scales were easy to be removed. Over the period of the next twenty-five months scale thickness increased only slightly to about 5-6 mm. A first description of scale composition and mineralogy was described by Haas-Nüesch et al. 2018.



Figure 9 Scale deposit inside the monitoring pipe with injection of the barium sulfate antiscalant (Reference Inhibitor) after 4 months exposure.

The combined application of the antiscalant Reference 1 in combination with the corrosion inhibitor Additive 3 showed excellent results in scale mass reduction to nearly 60-70%. Scale thickness after 4 months was <1 mm, Figure 10. Morphology of the scale was very different in comparison to the injection of solely Reference 1. The scale was also easy to remove but very brittle and paper like.



Figure 10: Scale deposit inside the monitoring pipe with injection of the barium sulfate antiscalant (Reference Inhibitor) in combination with the corrosion inhibitor (Additive 3) after 4 months exposure.

Since scale mass reduction is one of the most important objectives of the SUBITO research project, the results from both geothermal sites are summarized in Figure 11.

Antiscalants which focus only on barium sulfate mitigation produce the highest scale masses, see Ref. 1 and Ref. 2. All tested antiscalants which combined properties for barium sulfate and lead sulfide mitigation in one single product decreased total scale mass, see Inh. 1, Inh. 10 and Inh. 11. Best results were obtained by injecting two products, barium sulfate antiscalant with corrosion inhibitor. Here, the application of

Reference 1 in combination with Additive 3 decreased the total scale mass to nearly 60% in comparison to the application of only Reference 1 at the geothermal power plant at Insheim.

Analysis of the elemental composition and the mineralogical characterization are still ongoing but in a first conclusion it can be stated that filming agents like corrosion inhibitors can be efficiently used for scale mitigation in geothermal plants of the URG. It was observed that barium sulfate formation increased during combined injection of Inhibitor 11 with Additive 4.

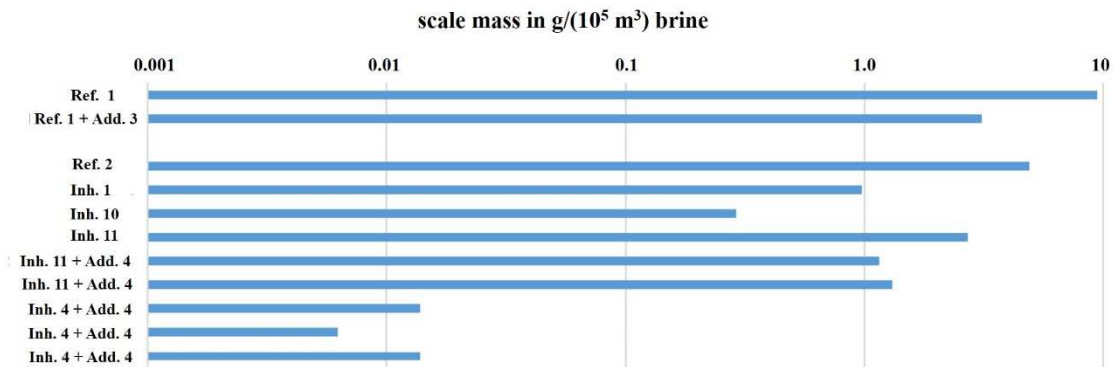


Figure 11: Evaluation of scale formation as a function of accumulated brine volume and deposited scale mass (logarithmic scale). Ref: barium sulfate antiscalant. Inh: “Sulfide-Inhibitor”. Add.: corrosion inhibitor.

3.4 Corrosion Monitoring

The injection of corrosion inhibitors in combination with barium sulfate antiscalants did not only reduce the total mass of scalings it also reduced the recorded incidents of localized corrosion to zero. In 2017 and 2018, corrosion related maintenance operations were frequently required. After start of the corrosion inhibitor injection in the 3rd Quarter of 2018 no corrosion maintenance was required and no leakages were recorded, Figure 12.

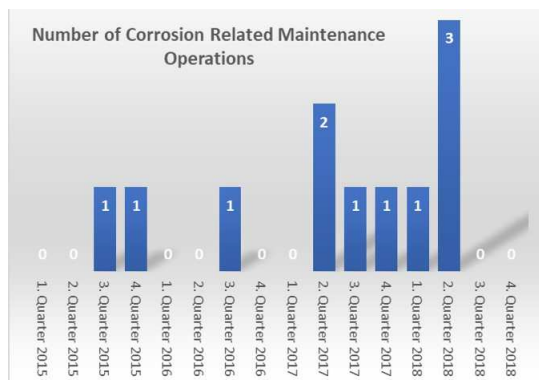


Figure 12: Number of corrosion related maintenance operations at the Insheim Geothermal Power Plant from 2015 to 2018.

Corrosion coupons were exposed to the geothermal brine at Insheim during the reference operation and during the test of the corrosion inhibitor. Uniform corrosion was generally very low and pitting corrosion

was not detected. Scales covered all surfaces of the coupons and of the coupon holder after exposure to brine which was treated solely with barium sulfate antiscalant. As soon as the corrosion inhibitor was added to this treatment the total amount of scales decreased which confirmed the results of the scale sampling from the monitoring pipe, Figure 13.



Figure 13: Top: Coupons after exposure to brine treated only with barium sulfate antiscalant. Bottom: Coupons after exposure to brine treated with barium sulfate antiscalant and corrosion inhibitor.

4. CONCLUSION AND OUTLOOK

The partners of the research project SUBITO realized an extensive study of different antiscalants and corrosion inhibitors in the laboratory and on-site of two different geothermal power plants at the German and the French side of the Upper Rhine Graben.

Brine/Inhibitor incompatibility needs to be confirmed for high temperature and low temperature conditions even if retention time in the feed line is very short.

Chemicals for scale and corrosion mitigation can interact with each other when more than one product is applied. In the best but very rare case the efficiency of one or both products increase as it was observed during the laboratory test of Comb 1. Unfortunately, it happens more often that the antiscalant efficiency is reduced by the simultaneous injection of a corrosion inhibitor. Lab tests proofed this phenomenon for Comb 2 and Comb 3 and barium sulfate scale increased in mass during the field application of an antiscalant in combination with a corrosion inhibitor.

The formation of metal-rich deposits can be retarded by antiscalants which inhibit barium sulfate and sulfide in one product. However, the best results were obtained by using two very different products at the same time. One powerful antiscalants for barium sulfate mitigation and a corrosion inhibitor. The presence of elemental lead indicated strongly the influence of electrochemical reactions on scale formation in the URG and the scale mass reduction of probably more than 60% due to the application of a corrosion inhibitor proofed this theory.

Despite the good results of the first tests, the remaining scale mass is still high for efficient performance of geothermal power plants in the URG and needs to be further reduced. Here, dose rate adjustment could be one way.

The SUBITO project showed clearly how difficult it is today for the power plant operators to select an appropriate product or better product combinations which could possible lead to an efficient and feasible brine treatment in the URG. Today it was possible thanks to an intense brine and scale monitoring but also this work is still in progress.

The mitigation of exotic scales which contains elements like Pb, As and Sb as main components requires a much better understanding of the mode of operation of the different products and their various constitutes with the geothermal brine but also with other products when applied in product combinations.

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