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Corrosion inhibitors and lubricants characterization using the Rock-Eval® Shale Play™ method: Case studies to determine the origin of geothermal scales

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A B S T R A C T

The main idea of this work is to propose an alternative analytical methodology to determine the origin of geothermal scales via thermal degradation experiments using the Rock-Eval® Shale Play™ method. The methodology consists of recording the specific Sh0, Sh1 and Sh2 parameters measured during both the vaporization and pyrolysis steps between 100 and 650 °C. Measurements are first performed on solid geothermal scales to better quantify the bulk total hydrocarbon compounds released during the thermal degradation, and then analyses are performed on both corrosion inhibitor and lubricant samples. Rock-Eval® Shale Play™ parameters obtained for each investigated sample are then compared to determine if the corrosion inhibitor or the lubricant can be also considered as the main cause of the scale formation in the investigated geothermal power plants. Results indicate that there is no apparent correlation between the corrosion inhibitor and the solid scale (case study 1). However, it seems that the use of lubricants could be associated to scale formation processes in the surface installations of geothermal power stations (case study 2).

1. Introduction

As a source of an always-available renewable energy, geothermal systems have been widely used to heat building since the late 1800s and to provide electricity since the 1900s. However, installed geothermal power stations only accounts for about 1% to the worldwide electricity mix (e.g. Brommer, 2018). Geothermal power plants use steam from high temperature wells to produce both electricity and hot water (e.g. Karlsdottir, 2012). Geothermal environments therefore require high-temperature hydrothermal resources, and advanced technologies to drill geothermal wells. One problem in the development of geothermal energy is the scale formation onto the surface of the geothermal wells or equipment like pipelines or heat exchangers (e.g. Andritsos et al., 2002; Ocampo-Díaz et al., 2005; Mundhenk et al., 2013; Klapper et al., 2016). In general, the formation of scale deposits in geothermal systems is mainly controlled by brines naturally concentrated in dissolved salts, the presence of other geothermal fluids, or the use of chemical additives during production operations. The scale precipitation is mainly due to several factors such as decreases of the temperature (the solubility for some salts decreases when the temperature drops), pressure reductions which cause flashes of dissolved gas (e.g. flash of CO_2 which increases the pH and can precipitate certain minerals) or a gradual increase in the dissolved iron content by corrosion of the tubing. These conditions are favorable to scale precipitation that can be found in wells, flash balloons and/or heat exchangers. In other words, according to Karlsdottir (2012), scaling occurs when minerals dissolved in geothermal fluid precipitate from the liquid and deposit onto the surface of the geothermal wells and equipment (due to a change in pressure, temperature, or pH value, which disturbs the equilibrium of the system). Thus, geothermal systems can undergo corrosion, scaling, or both simultaneously. In general, the most common classes of geothermal scales are (1) silica and silicates, (2) carbonates, and (3) sulfide compounds. However, it is also possible to detect organic deposits related to liquid hydrocarbons still present in the sedimentary basin. An alternative methodology could be necessary to better characterize the corresponding organic part of geothermal scales.

Furthermore, it should be noticed that geothermal fluids can be present in liquid, steam, or gas phases depending of the pressure and the temperature. These fluids are in direct contact to the material used in high-temperature geothermal wells and equipment, inducing corrosion

problems in geothermal power plants (e.g. Karlsdottir, 2012).

Chemical additives such as corrosion inhibitors and lubricants are thus used to protect all metallic surfaces against corrosion, cooling, and cleaning in geothermal plants (e.g. Ahmed and Nassar, 2011). Amongst others, these compounds have been characterized by fluorescence spectroscopy and chemometric techniques (e.g. Mbogning Feudjio et al., 2021).

During last years, geothermal scales or solid deposits have been widely investigated at the laboratory using different available techniques such as: X-ray diffraction (XRD), chemical elemental analysis using ICP-AES or ICP-MS (Inductively Coupled Plasma Atomic Emission Spectrometry or Mass Spectrometry), SEM-EDX (Scanning Electron Microscopy and Energy Dispersive X-ray fluorescence), XPS (X-ray photoelectron spectroscopy), EA-IRMS (Isotope Ratio Mass Spectrometry coupled to an Elemental Analyser), Raman spectroscopy and XANES (X-ray Absorption Near Edge Spectroscopy) (e.g. Peralta et al., 1996; Haas-Nüesch et al., 2018; among others).

The Rock-Eval® is an open-system pyrolysis and oxidation device that is widely used to characterize the thermal degradation of the sedimentary organic matter using different thermal methods. This technique has been mainly developed to investigate the petroleum potential of source rock in oil and gas exploration campaigns (e.g. Espitalié et al., 1977, 1986, 1987; Lafargue et al., 1998; and others). In 2014, in order to improve the source rock characterization, a specific Rock-Eval® Shale Play™ method (Fig. 1) was developed providing a dedicated pyrolysis program and associated parameters for evaluation of in-situ liquid hydrocarbon contents (e.g. Romero-Sarmiento et al., 2014; 2016a; b and references therein). This method provides 3 key-parameters such as Sh0, Sh1 and Sh2 peaks (Fig. 1). These parameters each represents a specific pool of organic compounds with unique thermal reactivity and chemical composition. Concerning the hydrocarbon composition detected by each Rock-Eval® Shale Play™ parameter, it has been previously demonstrated that low-to-medium molecular weight thermovaporized hydrocarbons ($<C_{20}$) are the main organic compounds released at the temperature range corresponding to the Sh0 parameter (100 °C–200 °C), whereas medium-to-high molecular weight hydrocarbons (C_{10}–C_{30} thermovaporized organic compounds) are predominant components thermally released in the temperature range corresponding to the Sh1 parameter (200 °C–350 °C; Fig. 1; Romero-Sarmiento et al., 2016b).

Recently, the Rock-Eval® device has been also used to both identify and quantify the presence of plastics in sediments (Romero-Sarmiento et al., 2022). In this manuscript, in order to expand the field of application of the Rock-Eval® Shale Play™ method, the analytical methodology was tested, for the first time, for the characterization of different solid and liquid samples that could be present in worldwide geothermal power installations as follow: (1) solid deposits or geothermal scales, (2) lubricants and (3) corrosion inhibitors.

It seems that open-system pyrolysis techniques like Rock-Eval® device could be also used as a fast and recently solution to characterize corrosion inhibitors, lubricants, and geothermal scales showing organic components. The application of the specific Rock-Eval® Shale Play™

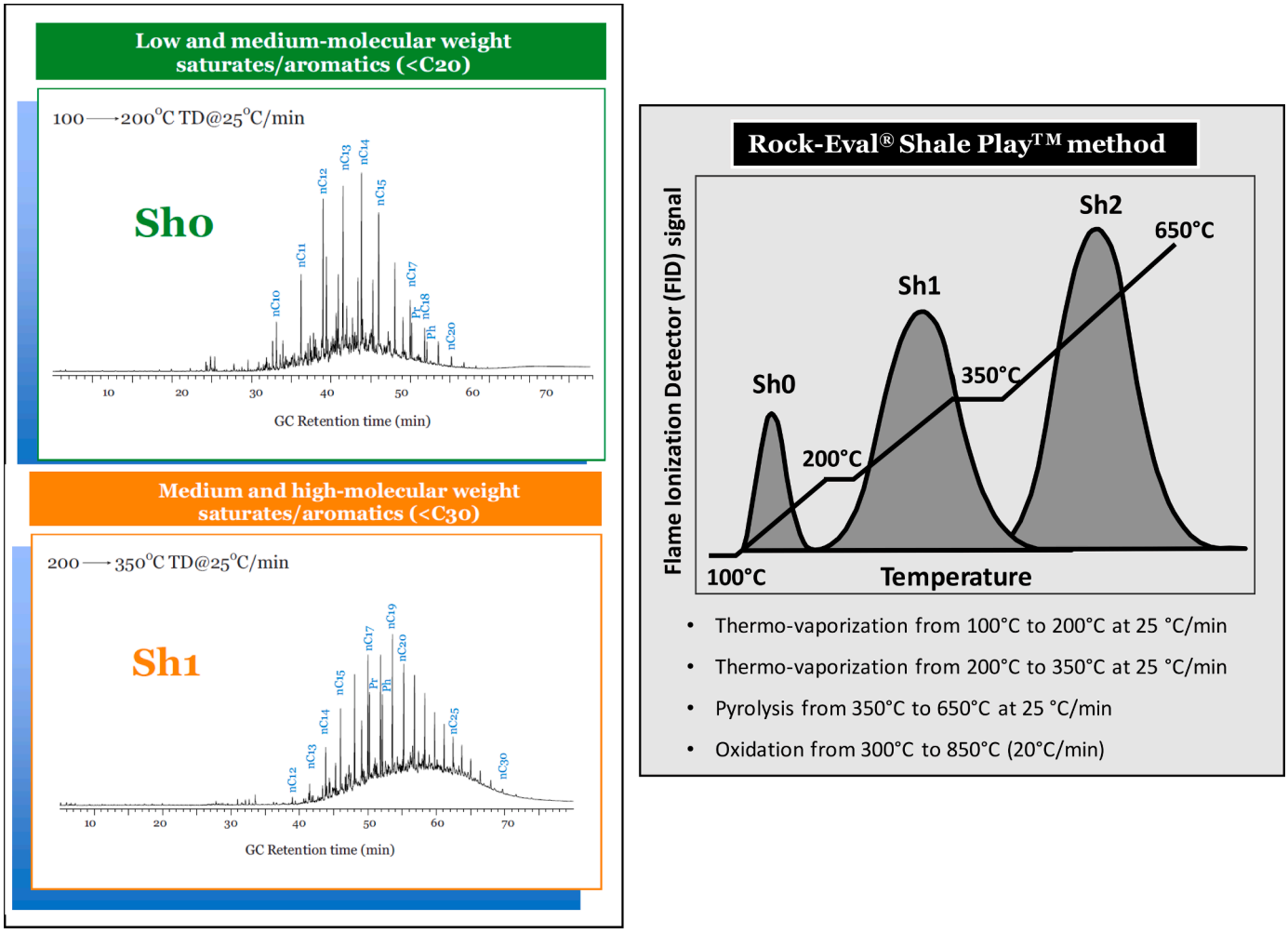

Fig. 1. Rock-Eval® Shale Play™ thermogram describing the specific thermovaporization, pyrolysis and oxidation conditions as well as the obtained parameters: Sh0, Sh1, Sh2 (modified from Romero-Sarmiento et al., 2014; 2016a,b; Romero-Sarmiento, 2019).

method to characterize these 3 kind of samples is the main idea of the present work, attempting to distinguish if the origin of solid deposits in geothermal power plants could be also associated to the frequently use of chemical additives like lubricants and corrosion inhibitors.

In this work, a comparative approach is proposed to compare the specific Rock-Eval® Shale Play™ parameters (Sh0, Sh1 and Sh2 peaks) obtained from solid deposits with those obtained from both corrosion inhibitors and lubricants. The purpose of this work is to investigate if t the corrosion inhibitor or the lubricant could be also responsible of these solid scale formations in geothermal power plants.

2. Materials and methods

2.1. Materials: corrosion inhibitor, lubricant, and geothermal scale samples

In this work, two case studies were selected from two different geothermal sites located in the eastern part of the Paris Basin, Ile-de-France region (France). Both geothermal sites are currently exploiting the carbonate Dogger reservoir which is the main geothermal aquifer used in the Paris region of France (1500–2000 m deep). Recently, geothermal operations conducted in the Dogger aquifer employ a heat pump to enhance the available geothermal resource. The local reinjected fluid temperature depends on the production temperatures (55 – 85 °C; Marty et al., 2020)

In the Case study 1, a manufacturing corrosion inhibitor used to protect the geothermal installation 1 as well as a solid deposit scale 1 deposited in the heat exchanger were investigated. The corrosion inhibitor is mainly composed by alkyl C_{10}—C_{16} dimethyl-benzyl-ammonium chloride whereas the solid deposit scale 1 is mostly composed by iron sulfides and silicates but also organic components.

For the Case study 2, a solid deposit scale 2 carefully precipitated from the produced brines as well as an oil-based lubricant were selected. The lubricant sample is mainly composed by oil whereas the solid deposit scale 2 is mainly composed by an organic-rich matrix (19.3 wt.%) previously characterized by elemental analysis.

2.2. Methods: Rock-Eval® Shale Play™ method and protocols

A Rock-Eval® 6 device operating at the IFP Energies Nouvelles (France) was used here. The Rock-Eval® 6 device is equipped with two ovens dedicated for pyrolysis and combustion processes, respectively. During the pyrolysis step, all hydrocarbon (HC) compounds generated are monitored by a Flame Ionization Detector (FID) whereas the non-hydrocarbon compounds like carbon monoxide (CO) and carbon dioxide (CO_2) released during both pyrolysis and oxidation stages are monitored by an infra-red (IR) detector (Espitalié et al., 1986; Lafargue et al., 1998; Behar et al., 2001). In this work, the Rock-Eval® Shale Play™ method described in Romero-Sarmiento et al. (2014; 2016a,b) was tested (Fig. 1). This specific method is characterized by a starting pyrolysis step at low temperature (100 °C) then the oven temperature increases from 100 °C to 200 °C at 25 °C/min. This last temperature is maintained for 3 min. The temperature is raised again from 200 °C to 350 °C at 25 °C/min. A plateau of 3 min is imposed at 350 °C and finally the pyrolysis temperature increases from 350 °C to 650 °C at 25 °C/min (Romero-Sarmiento et al., 2014; 2016a,b; Fig. 1). Concerning oxidation conditions, the oxidation temperature increases from 300 °C to 850 °C at 20 °C/min. Fig. 1 also illustrates the main Rock-Eval® Shale Play™ parameters used in this study. The *Sh0* parameter provides the quantity of hydrocarbon compounds released during the thermal vaporization step from 100 °C to 200 °C. The *Sh1* parameter measures the quantity of hydrocarbon compounds released during the thermal vaporization step from 200 °C to 350 °C and the *Sh2* parameter the quantity of hydrocarbon compounds released during the final pyrolysis step from 350 °C to 650 °C.

For geothermal scales characterization, the analytical procedure proposed in this study is described as follow:

(i) First, the solid scale 1 directly sampled from heat exchanger filters was cleaned, dried, and crushed to obtain a pulverized sample. The solid scale 2 recovered from geothermal brines was filtered, dried, and crushed to obtain a pulverized sample as well. Corrosion inhibitor and lubricant samples were analyzed on "as received" samples meaning that no analytical preparation protocol was applied.
(ii) Triplicate Rock-Eval® Shale Play™ analyses were carried out on 30 mg of each powdered scale sample whereas aliquots of corrosion inhibitors and lubricants around 5–15 mg were diluted with silica in Rock-Eval® crucibles in order to prevent saturations of both FID and IR detectors. These measurements were first used to define the corresponding Rock-Eval® thermograms for each investigated sample.
(iii) Finally, the specific Rock-Eval® Shale Play™ parameters (Sh0, Sh1 and Sh2 peaks) obtained from solid deposits are compared with those obtained from both corrosion inhibitor and lubricant samples to determine the origin of geothermal scale formations.

The methodology tested in this work was mainly focused on previously works performed by Romero-Sarmiento and Ravelojaona (2019).

3. Results and discussion

3.1. Corrosion inhibitor, lubricant, and geothermal scale properties as a function of the artificial thermal degradation

Table 1 summarizes the Rock-Eval® Shale Play™ parameters obtained in this study. Each investigated sample shows specific FID signals as a function of the artificial thermal degradation using a Rock-Eva® device. These signatures can be considered as thermal characteristics for each corrosion inhibitor, lubricant and geothermal scale samples. Fig. 2 shows the Rock-Eval® Shale Play™ FID thermogram obtained from the investigated oil-based lubricant illustrating the distinct parameters associated to the artificial thermal degradation (HC signals). Regarding the FID thermograms, it has been previously demonstrated using thermal desorption – gas chromatography (TD-GC) measurements, that low-to-medium molecular weight thermovaporized hydrocarbons ($<C_{20}$) are the main organic compounds released at the Sh0 temperature range (100 °C–200 °C), whereas medium and high molecular weight thermovaporized compounds ($< C_{30}$) are the predominant components that are thermal released in the temperature range corresponding to the Sh1 parameter (200 °C–350 °C) (more details in Romero-Sarmiento et al., 2016b; Romero-Sarmiento, 2019). Results presented in Fig. 2 thus indicate that the investigated lubricant is composed of medium-to-high molecular weight hydrocarbons (C_{10}–C_{30} thermovaporized organic compounds) released in the temperature and time ranges corresponding to the Sh1 parameter (200 °C to 350 °C and 10 to 15 min). Furthermore, heavy molecular weight hydrocarbons ($>C_{30}$ organic compounds) are the most predominant components thermally released during the final pyrolysis step of the lubricant sample from 350 °C to 650 °C (Sh2 parameter).

In contrast, the investigated corrosion inhibitor (Fig. 3) is dominated by both low-to-medium molecular weight thermovaporized hydrocarbons ($<C_{20}$) that are the main organic compounds released at the temperature range corresponding to the Sh0 parameter (100 °C–200 °C) as well as by heavy molecular weight hydrocarbons ($>C_{30}$) thermally released in the temperature range corresponding to the Sh2 parameter (350 °C–650 °C; Fig. 3; Romero-Sarmiento et al., 2016b).

Finally, in this study, both geothermal scales are only characterized by the dominance of medium-to-high molecular weight hydrocarbons (C_{10}–C_{30}) released in the temperature and time ranges corresponding to the Sh1 parameter (200 °C to 350 °C and 10 to 15 min; example in Fig. 4– scale 2).

Table 1
Rock-Eval® Shale Play™ parameters from investigated samples.

Case study	Sample	Sh0 (mg/g)	Sh1 (mg/g)	Sh2 (mg/g)	TOC (wt.%)	MINC (wt.%)	HI (mgHC/gTOC)	OI ($mgCO_2$/gTOC)
1	**Scale 1**	1.24 ± 0.06	5.99 ± 0.15	0.51 ± 0.09	**1.59 ± 0.23**	0.71 ± 0.26	32 ± 1	156 ± 34
	Corrosion Inhibitor	113.23 ± 1.27	40.39 ± 1.44	99.93 ± 1.58	**21.54 ± 0.16**	0.27 ± 0.05	464 ± 4	17 ± 5
2	**Scale 2**	0.47 ± 0.05	132.73 ± 1.18	20.72 ± 1.09	**15.39 ± 1.03**	2.23 ± 0.56	135 ± 3	113 ± 1
	Lubricant	9.31 ± 4.40	310.06 ± 5.83	555.92 ± 15.30	**78.58 ± 2.46**	0.13 ± 0.12	708 ± 2	2 ± 1

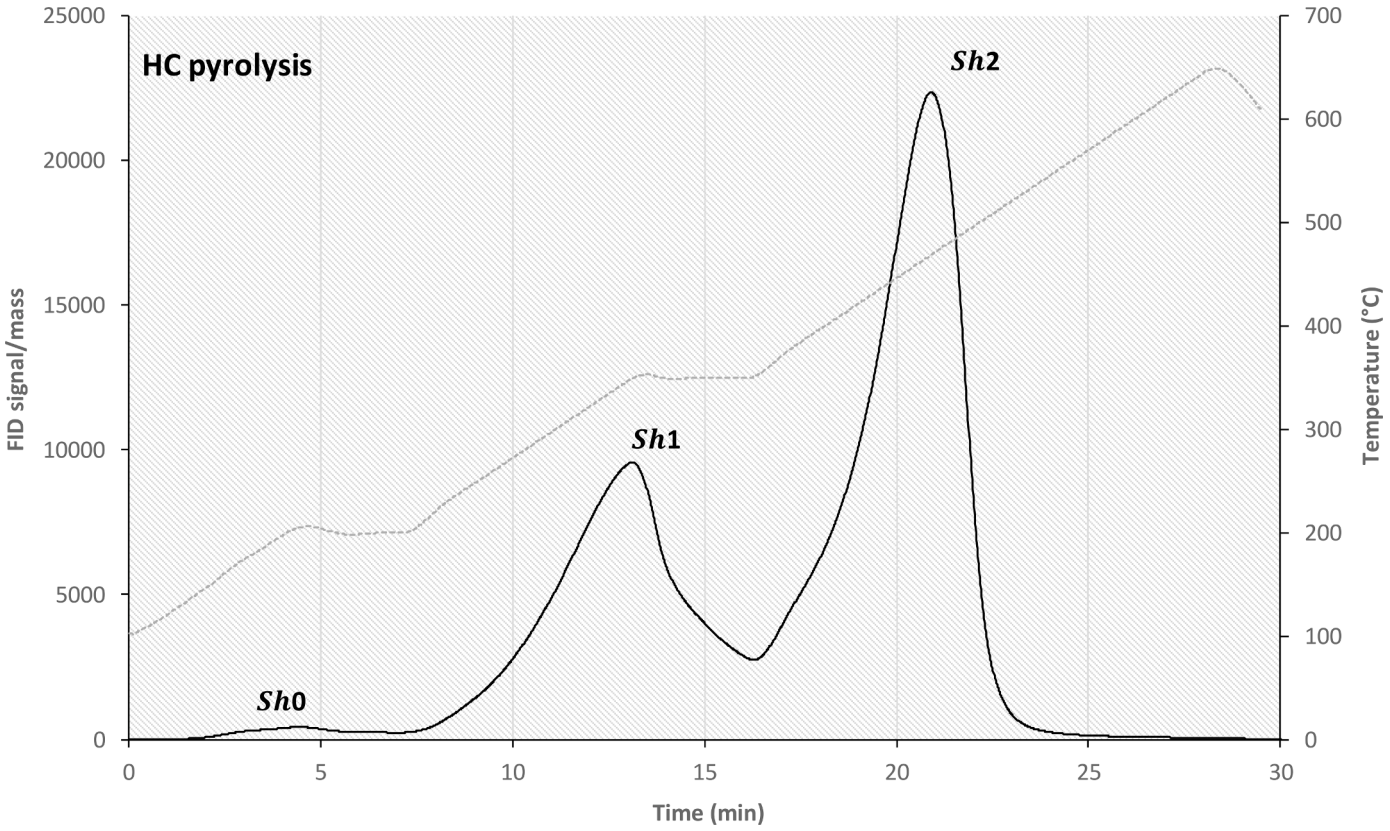

Fig. 2. Rock-Eval® Shale Play™ thermogram showing the distinct parameters associated to the thermal degradation of the investigated oil-based lubricant.

The total organic carbon (TOC) and the mineral carbon (MINC) contents of the corrosion inhibitor, lubricant and geothermal scale samples are also showed in Table 1. The TOC expressed as a weight percent, represents the amount of organic carbon presents in geothermal scales. This parameter can be also used to estimate the organic nature of the investigated solid scales.

3.2. Comparison between corrosion inhibitor, lubricant and scale samples: geothermal operation implications

The total quantity of organic compounds released from each analyzed sample and measured by the Rock-Eval® FID detector (Sh0, Sh1 and Sh2 peaks), was compared to evaluate if the chemical additives (e.g. corrosion inhibitors and lubricants) are responsible for the deposition of the solid scales present in geothermal installations. For the case study 1, Fig. 5A illustrates an example of the application of this methodology demonstrating that there is no apparent correlation between the investigated corrosion inhibitor and the solid scale. However, for the second case study, it seems that the lubricant could be associated to the scale formation in the geothermal power plants (Fig. 5B). For geothermal operations, this implies that this fast-thermal degradation method and this simple comparison approach can be also used like an alternative technique to distinguish the origin of geothermal scales.

To summarize, obtained results illustrate the potential of the Rock-Eval® device as a quick technique (~30 min open-system pyrolysis) to characterize the following samples: lubricants, corrosion inhibitors and solid deposits. It could be considered as an additional screening approach to provide complementary information to other methodologies such as Raman or XRD techniques. To evaluate in more detail the specific composition of these samples, it is recommended to perform advance analytical workflow including Py-GC–MS.

4. Conclusions

A quick analytical methodology using the Rock-Eval® Shale Play™ method was proposed here in order to identify the origin of scalings formed in geothermal sites. Sh0, Sh1 and Sh2 parameters can be used to characterize corrosion inhibitor, lubricant and geothermal scale samples. These parameters can be also considered as a useful tool to distinguish the possible correlation between the solid formed material and the chemical additives. In this study, after the application of corrosion inhibitors and lubricants, it was demonstrated that there is no apparent correlation between the corrosion inhibitor and solid scale formations (case study 1). However, results indicate that the use of lubricants could contributed to organic scale formation processes in the surface installations of geothermal power stations (case study 2).

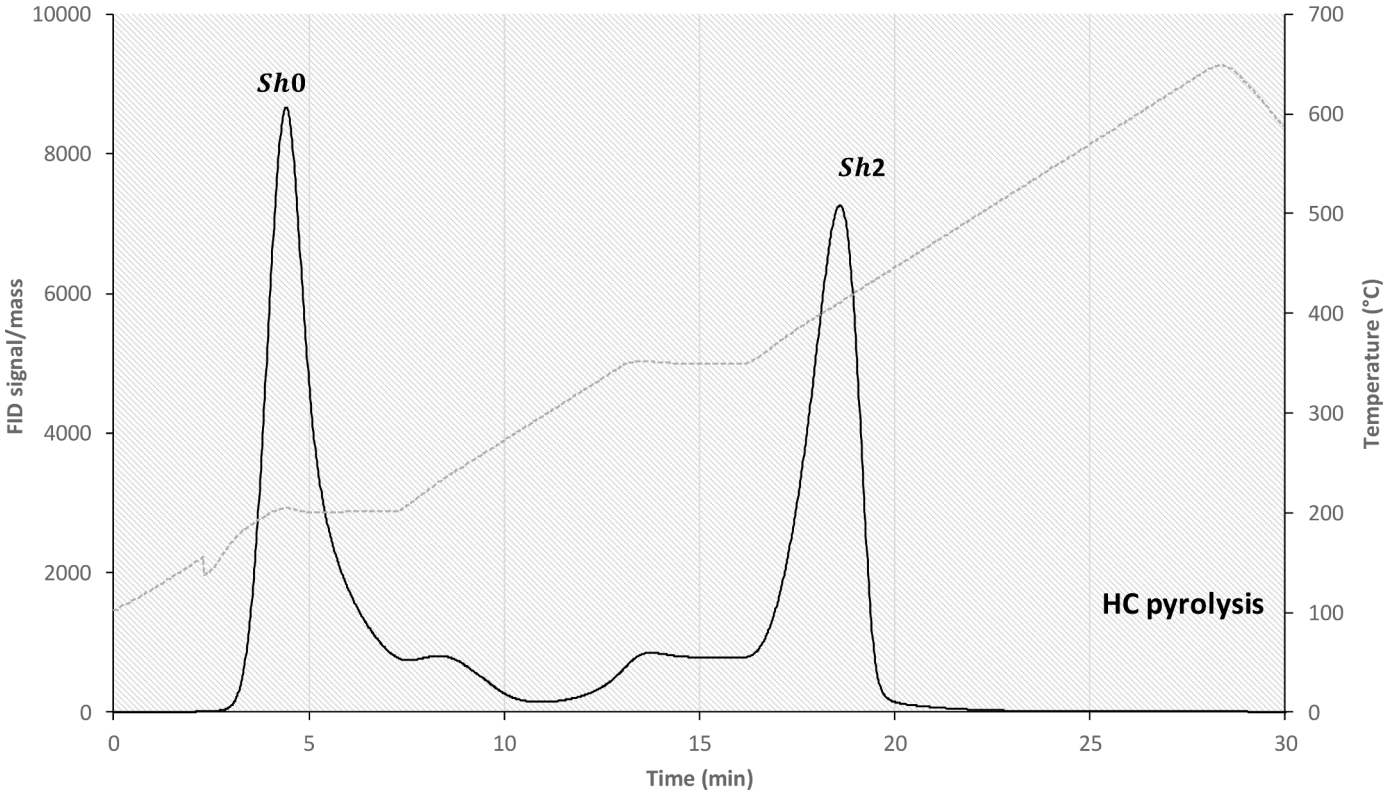

Fig. 3. Rock-Eval® Shale Play™ thermogram showing the distinct parameters associated to the thermal degradation of the selected corrosion inhibitor.

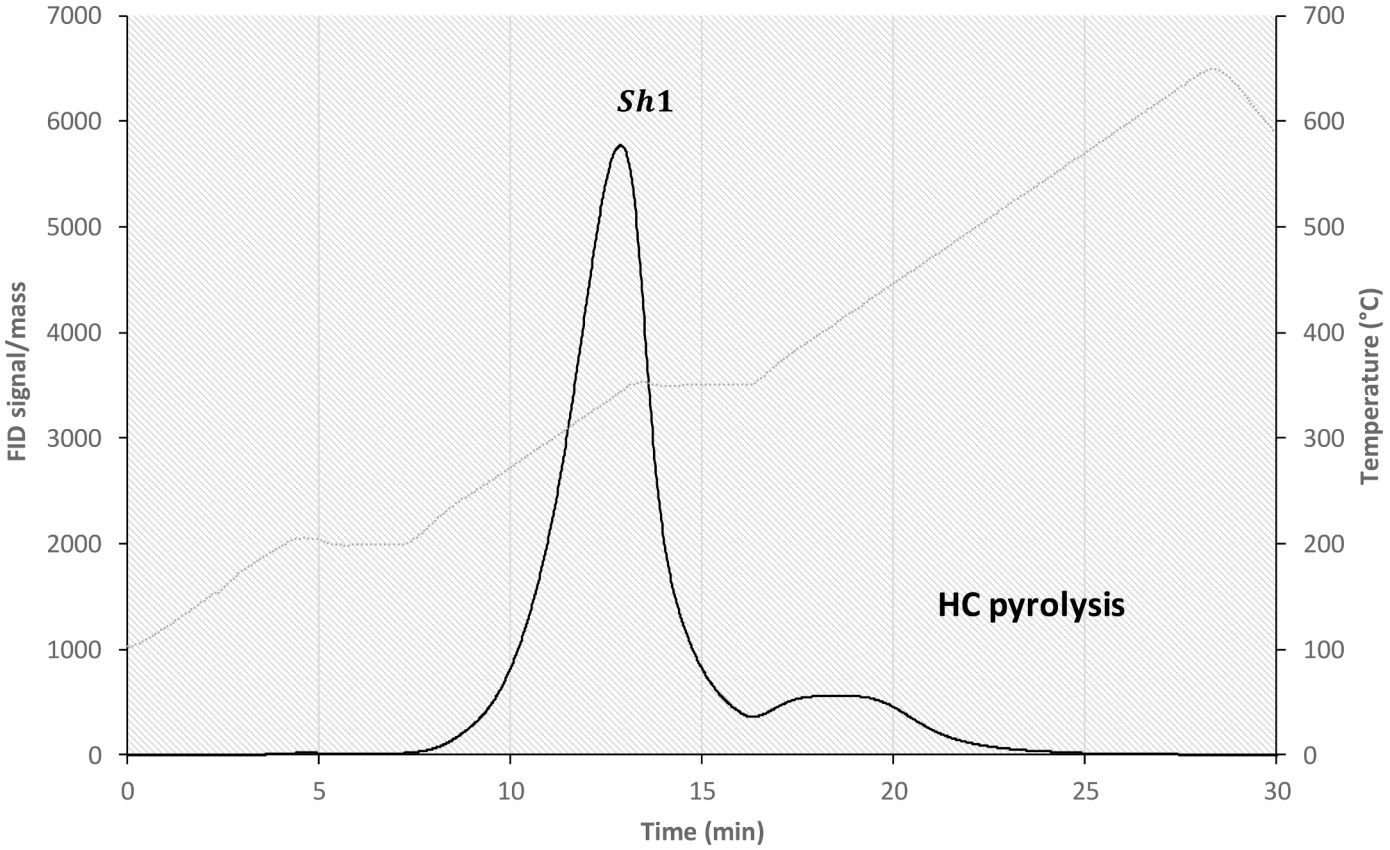

Fig. 4. Rock-Eval® Shale Play™ thermogram showing the distinct parameters associated to the thermal degradation of the selected geothermal scale (case study 2 – scale 2).

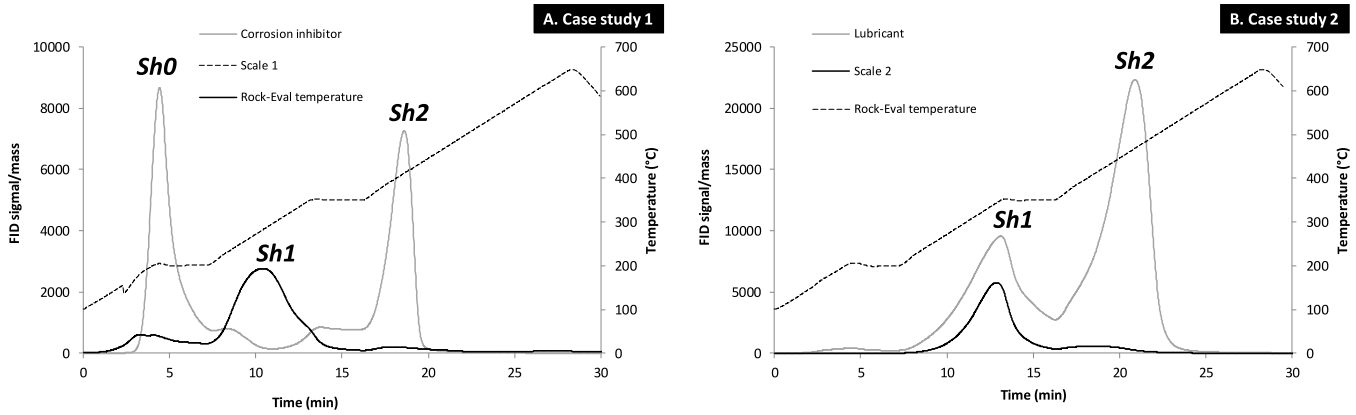

Fig. 5. Comparison of Rock-Eval® Shale Play™ FID thermograms showing the specific Sh0, Sh1, Sh2 parameters obtained from corrosion inhibitor, lubricant, and geothermal scale samples (case studies 1 versus 2).

Credit author statement

Maria-Fernanda Romero-Sarmiento: Conceptualization, Methodology, Investigation, Data curation, Writing- Original draft preparation. Herman Ravelojaona: Conceptualization, Methodology. Nicolas Maubec: Supervision.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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