

Article

Study on the Influence of Non-Metallic Inclusions on the Pitting Corrosion of API 5L X60 Steel

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Abstract: The most economical way to move liquids and gaseous hydrocarbons is by using pipelines. According to several international organizations and oil companies, the use of fossil fuels will continue in the following decades. For this reason, it is important to continue studying different corrosion mechanisms and their origins. One of the mechanisms that provoke small leaks, affecting pipeline hermeticity, is pitting corrosion. It is well-known that non-metallic inclusion dissolution can trigger pit nucleation. As pitting corrosion is recognized to be random in nature, it is also interesting to study the random nature of the inclusions present in API 5L X60 steel. Probability distributions commonly used to describe pitting corrosion characteristics are appropriate for studying inclusion characteristics. The size of inclusions plays a key role in pit nucleation because small inclusions tend to generate more defects, especially when these inclusions are compounds of MnS, and the steel is immersed in a corrosive solution. The results of this research work show that there is a close relationship between the random nature of pitting corrosion and inclusions.

Keywords: pitting corrosion; pipeline steel; non-metallic inclusions; probability density function; MnS



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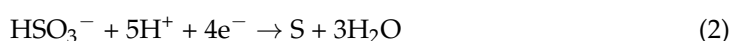
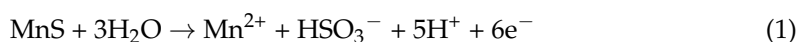


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1. Introduction

The importance of oil and gas pipelines is indicated by an estimated increase in their construction in the future due to factors such as the discovery of reserves in remote and landlocked locations, depletion of reserves near established markets, far distances from oil refineries and big cities and improvements in technology related to exploration and reserve production [1]. The IEA forecasts that oil consumption will remain above 85 million barrels per day, and the amount of natural gas consumed will remain above 4 trillion cubic meters until 2050 [2]. This means that pipelines will remain relevant for at least the first half of the 21st century. Other issues important to mention when oil and gas pipelines are studied are their safety and reliability. For example, these structures typically suffer from third-party damage, corrosion and construction defects, natural hazards, operational errors and material imperfections [3,4]. At present, aging of pipeline infrastructure is one of the main problems confronting the oil and gas industry. According to a study presented by Mojtaba Mahmoodian and Chun Qing Li [5], 50% of the USA's oil and gas pipeline network is over 40 years old. In Russia, 20% of the country's oil and gas system is almost

at the end of its design life, and it is expected that, at the beginning of the 2030s, half of their pipelines will be at the end of their design life. In these aged structures, different corrosion mechanisms threaten their mechanical integrity [3,6]. The strength of a pipeline can be impacted by corrosion-induced reductions in pipe wall thicknesses [7,8]. In the specific case of Mexican pipelines, the mechanism that causes the greatest threat to pipeline integrity is pitting corrosion, provoking a substantial number of leaks [9]. To estimate the damage provoked by this mechanism, pitting corrosion has been modeled using statistical techniques such as non-linear regression and Markov chains [10,11]. Likewise, the impact of the growth of these corrosion defects has been studied to determine pipeline reliability [12]. There are some theories regarding the origin of pitting corrosion: one is that pits occur when the oxygen adsorption point on the surface is replaced by halide ions (e.g., chloride ions); another theory is that the radius of chloride ions is small, and therefore, ions can travel through the passivation film. The generated ions provoke the membrane to maintain a current density at a specific point, causing the cations to move. When the electric field at the membrane–solution zone reaches a critical value, pitting corrosion can occur [13]. Another possible cause of the origin of corrosion defects caused by the pitting mechanism is the dissolution of non-metallic inclusions [13]. According to Smialowski et al. [13] sulfide inclusions are very effective in pit nucleation. Other works proposed the following mechanism of dissolution for MnS inclusions in steel [14–16]:



The steel from which oil and gas pipelines are made has not only high yield strength values but also high ductility [17]. This is the result of its chemical composition and the manufacturing process. However, in pipeline steels, one can find many types of inclusions besides MnS, such as Al_2O_3 , silicates and complex oxides [18,19]. The chemical composition of inclusions is strongly associated with the steelmaking process and demonstrates a key role in the localized corrosion process. Some research works indicated that pitting corrosion tends to start when non-metallic inclusions are dissolved [20,21]; others suggested that dissolution started on the base material matrix around the inclusions [22]. For example, Figure 5 from Reference [22] schematizes this process following studies involving EDX spectra and FEM. Other research works have studied the shape of inclusions because, in some cases, spherical inclusions tend to generate pits [23]. Another variable that exerts influence in inclusion dissolution to generate pits is the chemical composition of the environment [22–25].

After considering these observations from other research works, one can determine that the actual mechanism of pitting corrosion defects caused by inclusion dissolution is not totally well-understood. For this reason, in the present study, we studied the relationship between inclusion characteristics and pitting corrosion defect characteristics found in API 5L X60 steel (in the oil industry, the American Petroleum Institute designates the standard API 5L [17] for pipe steel specifications; particularly, X60 is designated to pipeline steel that exhibits at least 60 Ksi in yield strength) after being exposed to immersion tests. These characteristics were analyzed using statistical techniques in order to determine the predominant shape and type of inclusions. Likewise, we determined the probability density function that best fits both inclusion characteristics and pit characteristics after different immersion times.

2. Materials and Methods

2.1. Sample Preparation

The experimental procedure began with steel sample preparation. First of all, from a half-round steel pipe, cubic samples ($1\text{ cm} \times 1\text{ cm} \times 1\text{ cm} = 1\text{ cm}^3$) were mechanically cut. After this process, samples were mounted in acrylic blocks. The samples were ground

using 240, 320, 400 and 600 grit papers initially and then fine-polished using an Al_2O_3 suspension. Figure 1 schematizes this process:

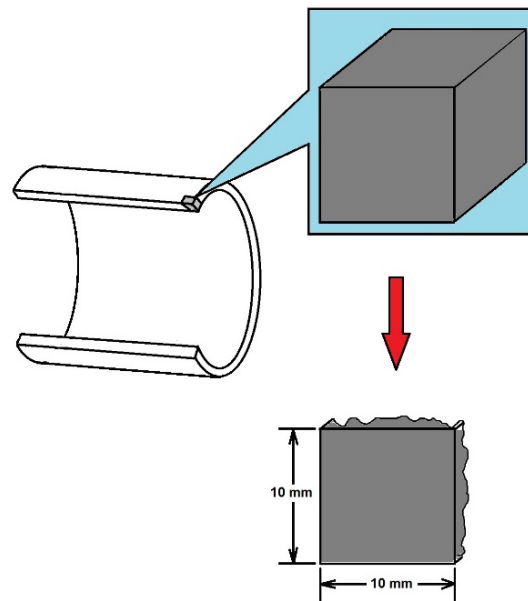


Figure 1. Schematic sample extraction from API 5L X60 steel pipe.

2.2. Microstructural Characterization and Chemical Composition

Table 1 shows the chemical composition of the API 5L X60 steel used in this research work. The chemical composition of API 5L X60 steel was obtained through Spark Atomic Emission Spectroscopy (SPECTRO Analytical Instruments GmbH, Kleve, Germany).

Table 1. API 5L X60 chemical composition.

Material	C	Mn	Si	P	S	Cu	Cr	Ni
Actual chemical composition	0.2	1.22	0.070	0.019	0.021	0.035	0.013	0.011
Specifications according to API 5L X60 (maximum allowable) [17]	0.26	1.4	-	0.03	0.03	-	-	-

The quantitative analysis of the microstructural characteristics of the API 5L X60 steel is presented in Table 2.

Table 2. API 5L X60 microstructural characteristics.

Ferrite	Pearlite	Grain Size [26]	
%	%	ASTM	μm
76	24	6	40

Figure 2 shows optical micrographs to illustrate the grain size and the percentages of pearlite and ferrite phases.

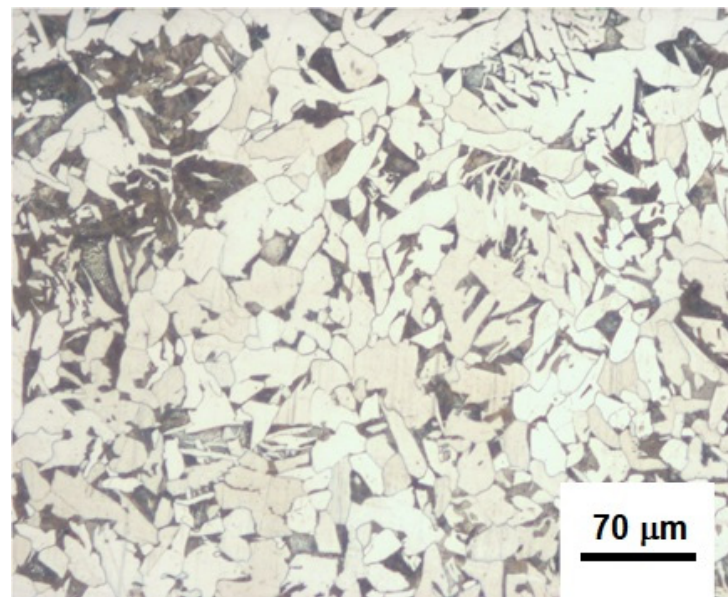


Figure 2. Microstructure of an API 5L X60 sample.

2.3. Non-Metallic Inclusions Content in API 5L X60 Steel

Non-metallic inclusion content was determined according to ASTM E45 [27]. For the sake of illustration, Figure 3 shows non-metallic inclusions found in the API 5L X60 steel.

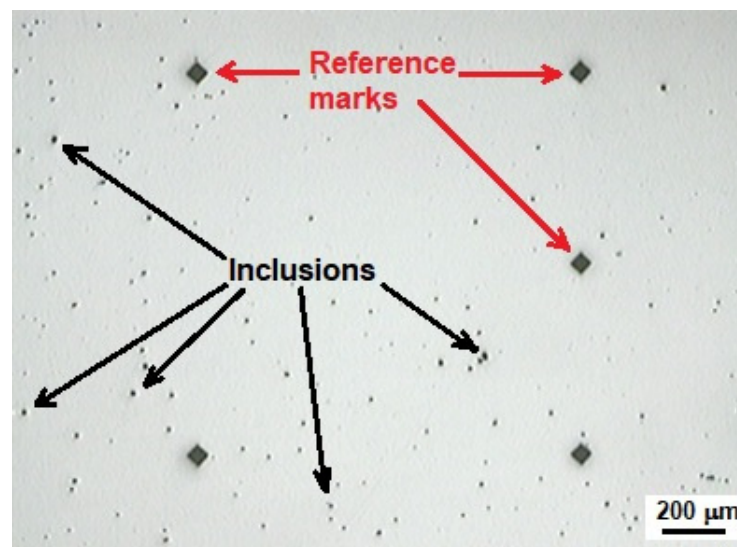


Figure 3. Typical non-metallic inclusions found in API 5L X60.

In addition to determining the density of inclusions, we estimated the chemical composition of the inclusions found using EDS. Likewise, we also estimated the shape of inclusions (using optical microscopy) (Olympus, Center Valley, PA, USA) in order to determine if circular inclusions have a greater influence on pit generation than elongated inclusions.

2.4. Immersion Test

Immersion tests were carried out to study the relationship between non-metallic inclusions found in API 5L X60 steel and pit generation and growth. The chemical compositions of solutions used in the immersion tests were similar to those found in a soil corrosiveness study in Mexico [28]. The chemical content of the prepared chemical solution was 16.84 ppm SO_4^{2-} and 289.5 ppm Cl^- , and pH = 4.35 at room temperature. The immersion

tests were carried out for two different periods of time. One battery of experiments was carried out over short periods of time (15, 30, 60, 180 and 360 min). In the other series of experiments, the immersion tests were accomplished after 1, 3 and 7 days. For illustration purposes, Figure 4 depicts the immersion test performed as part of the present work.

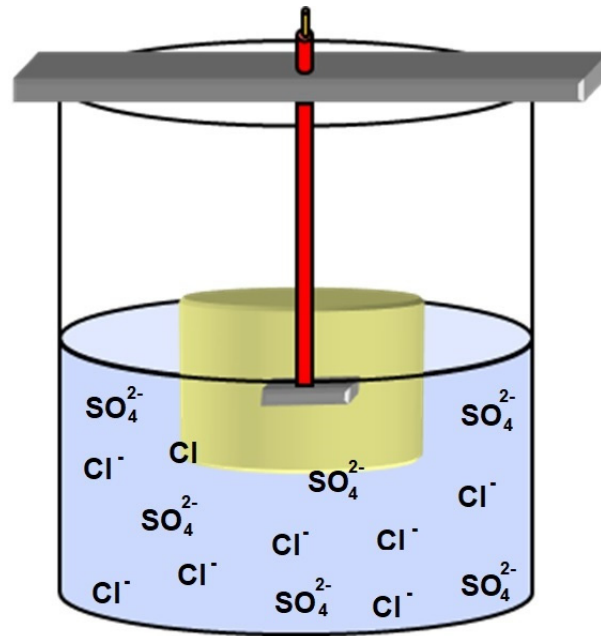


Figure 4. Schematic representation of the immersion test carried out.

2.5. Non-Metallic Inclusion Locations, Morphological Analysis and Characterization of Inclusions in API 5L X60 Steel

Non-metallic inclusion locational and morphological analyses were carried out using optical microscopy (Olympus, Center Valley, PA, USA). Otherwise, the chemical composition of inclusions was obtained using the SEM–EDS method. For the sake of illustration, an SEM image (obtained with the JEOL JSM-6300 (JEOL, Peabody, MA, USA)) of a non-metallic inclusion and EDS analysis (Thermo Scientific, Madison, WI, USA) of the chemical composition are shown in Figure 5.

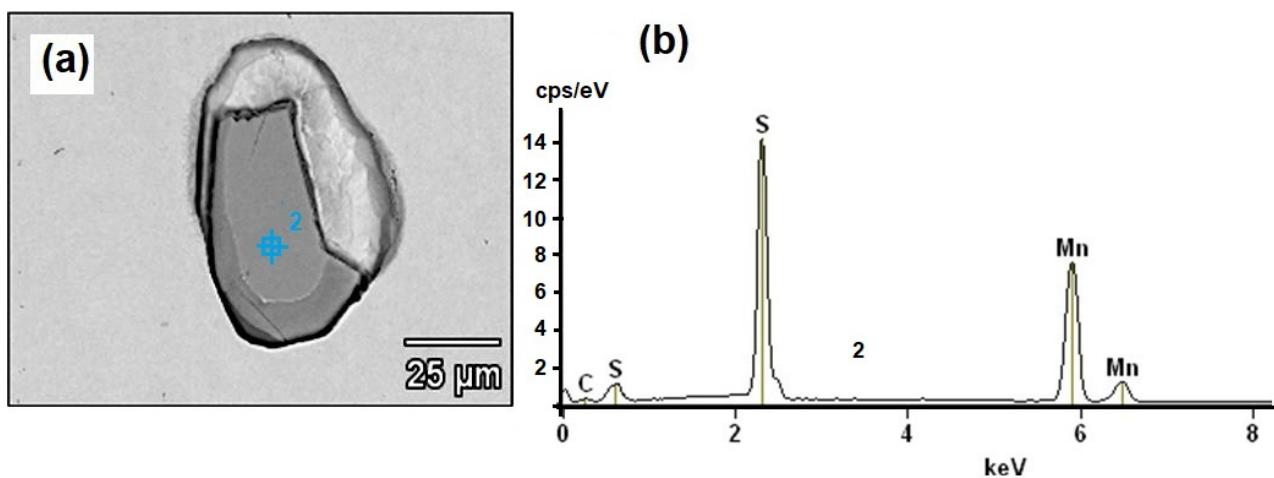


Figure 5. (a) Non-metallic inclusion SEM image and (b) chemical composition of the non-metallic inclusion, EDS spectra.

2.6. Inclusions' Statistical Characterization for Pit Generation

Non-metallic inclusions were characterized statistically by size (length and width), shape factor (width/length), chemical composition and area. Likewise, this information was related to data obtained through the characterization of pits generated after the immersion test over short periods of time (30, 60 and 180 min). The pit characteristics analyzed were depth and area. Additionally, we characterized pits by their depth generated during mid-term immersion tests (1, 3 and 7 days). All statistical analyses were performed using Wolfram Mathematica [29].

3. Results and Discussions

3.1. Statistical Analysis

The first step to characterize the non-metallic inclusions was to measure their length and width. With this information, one can calculate the shape factor (width/length). These data were obtained after analyzing one sample in three directions (rolling direction (RD), transverse direction (TD) and normal direction (ND)). Figures 6–8 show the histograms of length, width and shape factor, respectively. These histograms were obtained after characterizing all inclusions longer than 1 μm found in the analyzed sample.

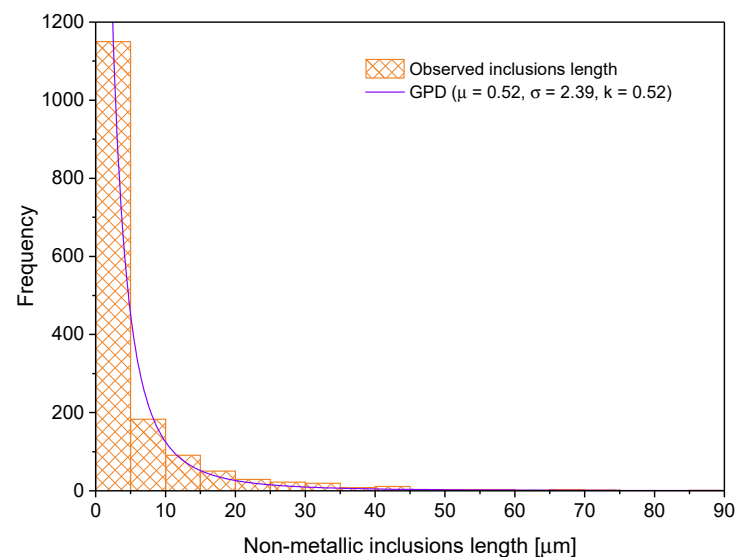


Figure 6. Non-metallic inclusion length distribution.

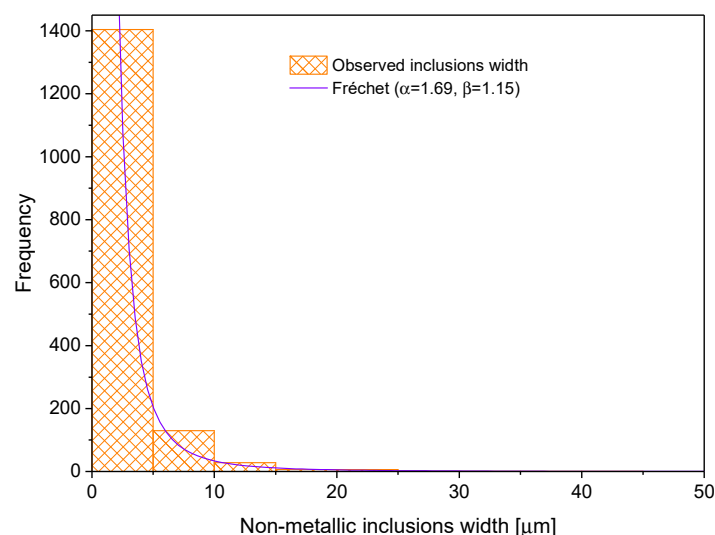


Figure 7. Non-metallic inclusion width distribution.

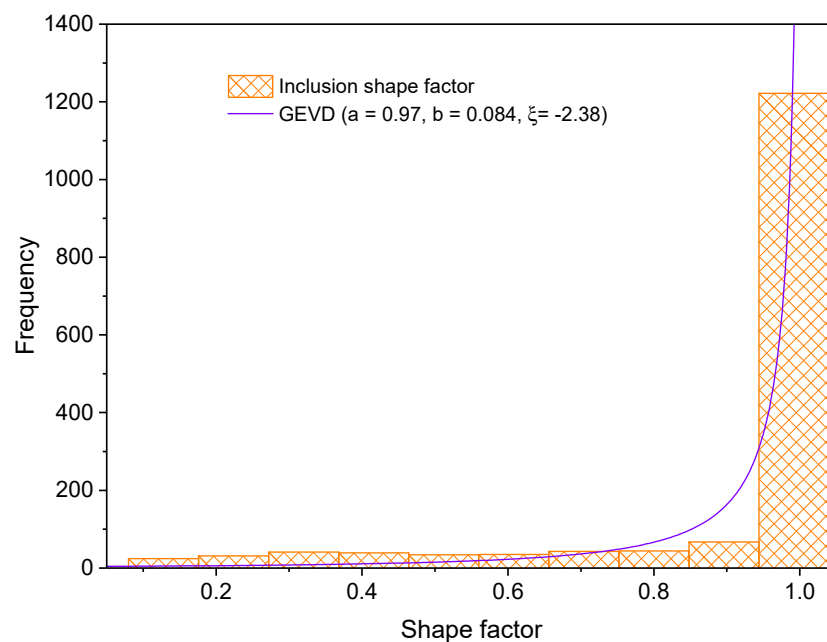


Figure 8. Inclusion shape factor histogram.

Comparing the results with others obtained in the literature [30–34], it is confirmed that, in all cases, the tendency is that the most frequent size of the inclusions is attributed to those inclusions that measure less than 10 μm in length.

These three histograms (Figures 6–8) were fitted to probability density functions (PDFs). The best choice was the PDF that satisfies the tests of Kolmogorov–Smirnov, Anderson–Darlin and chi-square. Regarding the random variable, inclusion length was fitted to a Generalized Pareto distribution (GPD). This function is represented by the mathematical expression indicated in Equation (3) [35].

$$f(x) = \frac{1}{\sigma} \left(1 + k \frac{x - \mu}{\sigma} \right)^{-1-1/k} \quad (3)$$

where μ , σ and k are the parameters of location, scale and shape, respectively. Concerning the inclusion width histogram, it was fitted to the Fréchet distribution, which is represented by Equation (4) [36].

$$f(x) = \frac{\alpha}{\beta} \left(\frac{\beta}{x} \right)^{\alpha+1} \exp \left(- \left(\frac{\beta}{x} \right)^{\alpha} \right) \quad (4)$$

where α and β are the shape and scale parameters, respectively. The shape factor histogram was fitted to the Generalized Extreme Value distribution (GEVD) expressed by Equation (5) [36].

$$f(x) = \frac{1}{b} \text{Exp} \left[- (1 + \xi z)^{-1/\xi} \right] (1 + \xi z)^{-1-1/\xi} \quad (5)$$

where $z = \frac{x-a}{b}$, a is the location parameter, b is the scale parameter and ξ is the shape parameter.

After this analysis, it is feasible to affirm that inclusion shape characteristics in API steel can be studied as random variables. This could help to estimate inclusion shape characteristics in pipelines by using theories such as return period [37]. The average non-metallic inclusion density is 160.5 inclusions per cm^2 .

After EDS analysis of the studied non-metallic inclusions' chemical composition, it was found that the vast majority of them were compounds of MnS (about 74%). Silicates were the second most frequent type of inclusions (mainly SiO_2 and CaOSiO_2), accounting for about 12% of the total. The remaining inclusions were compounds of Al_2O_3 , $3\text{CaO} \cdot \text{Al}_2\text{O}_3$,

$\text{CaO}\cdot\text{Al}_2\text{O}_3$, $\text{CaO}\cdot 2\text{Al}_2\text{O}_3$, $\text{CaO}\cdot 6\text{Al}_2\text{O}_3$, FeSiC , FeSiO , AlCaO , AlMgCaO , etc. Figure 9 depicts the distributions of the non-metallic inclusions found.

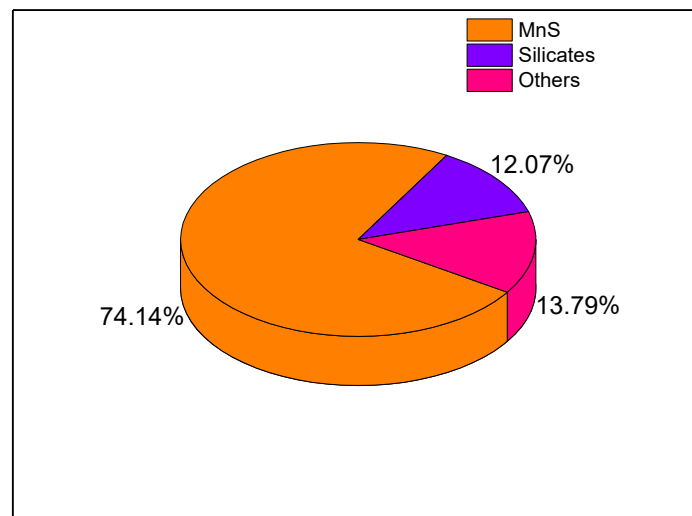


Figure 9. Non-metallic inclusions found in the API 5L X60 steel studied.

3.2. Role of Non-Metallic Inclusions in Pitting Corrosion

To understand the relationship between inclusions and pitting corrosion, we carried out immersion tests in a chemical solution as described in the previous section. In the first set of experiments, the immersion tests were performed for 30, 60 and 180 min. Once the microstructure was revealed (using a Nital solution) in each of the specimens, we proceeded to take micrographic sequences of the entire metallic surface (exposure area) in order to produce micrographic backups of each of the samples before micro-testing corrosion and to document their state before the immersion test.

After taking all the micrographs for a corresponding state, the micrographs taken at $50\times$ were stitched together in consecutive order to produce an image of the entire test area. The micrographs taken at $100\times$ were used to carry out microstructure and inclusion analysis, and $200\times$ images were used to learn more about the characteristics of the areas surrounding the micro-pits that were generated after the immersion test. Figure 10 illustrates the micrographic compositions corresponding to a test for a given exposure time. For the sake of exemplification, Table 3 shows the number of inclusions found after a certain immersion time.

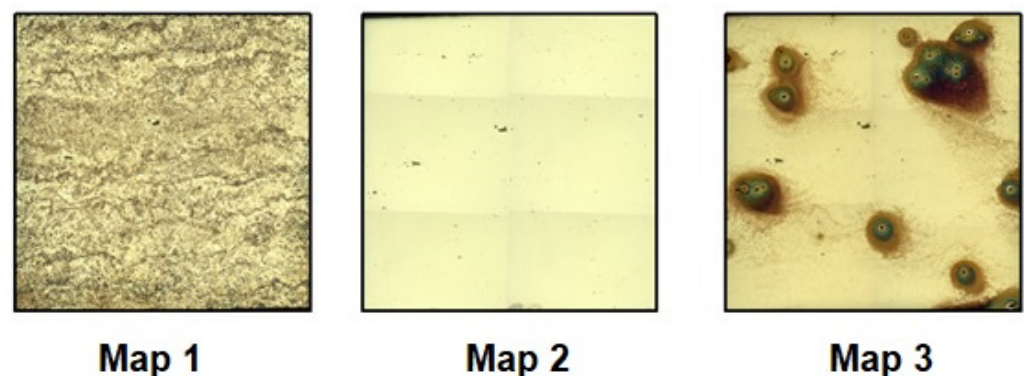
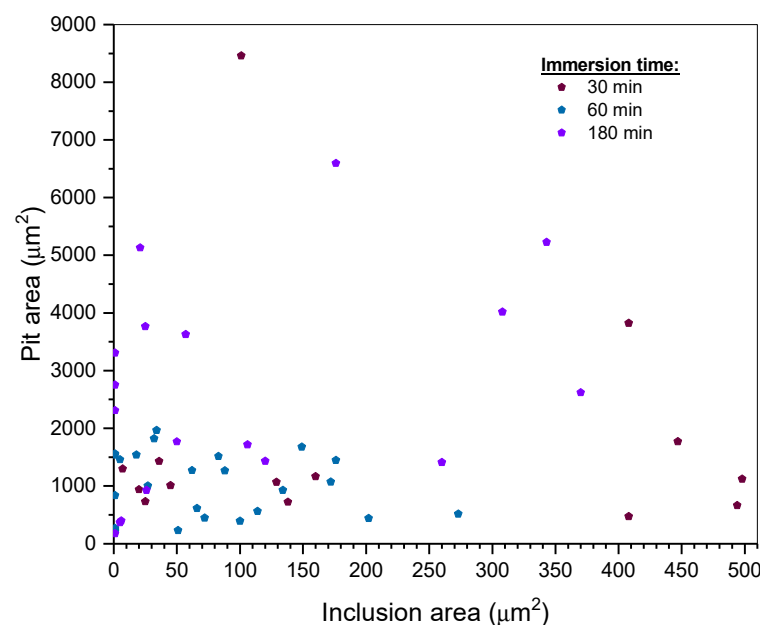


Figure 10. Mapping process to analyze the relationship between inclusions and pit nucleation.

Table 3. Summary of the relationship between total non-metallic inclusions and the place where the pits were nucleated.

Immersion Time (min)	Number of Inclusions	Number of Pits Generated	Sites of Nucleation (%)	Chemical Composition of Active Inclusions		
				MnS	Silicates	Others
30	600	14	2.33	14	0	0
60	717	25	3.49	16	3	6
180	610	18	2.95	13	1	4

For the sake of finding a relationship between inclusion area and pitting area, we plotted a scatter diagram as shown in Figure 11. One can notice a greater correlation between inclusion area and pit area when the inclusion area is smaller than $200 \mu\text{m}^2$ (Spearman's rho (correlation coefficient) equal to 0.50). The smaller the inclusion area is, the greater the probability of provoking pitting corrosion is.

**Figure 11.** Scatter plot of relationship between non-metallic inclusion area and pit area.

Inclusions that encouraged pit nucleation are named “active inclusions”. Figures 12 and 13 show histograms for active inclusion area and their shape factor. The active inclusion area histogram was fitted to the Generalized Pareto distribution (see Equation (3)). The histogram for active inclusion shape factor was fitted to Gumbel’s minimum value distribution represented by Equation (6) [36,37].

$$f(x) = \frac{1}{\sigma'} \exp(z' - \exp(z')) \quad (6)$$

where $z' = \frac{x - \mu'}{\sigma'}$, and μ' and σ' are the location and scale parameters, respectively. In Figure 11, we notice that inclusions with smaller areas tend to be more likely to cause pitting corrosion than larger inclusions. Otherwise, inclusions with a shape factor close to one tend to be more likely to provoke pits. However, it seems that elongated inclusions are more active than circular inclusions because the ratio of active elongated inclusions is higher than that of circular ones.

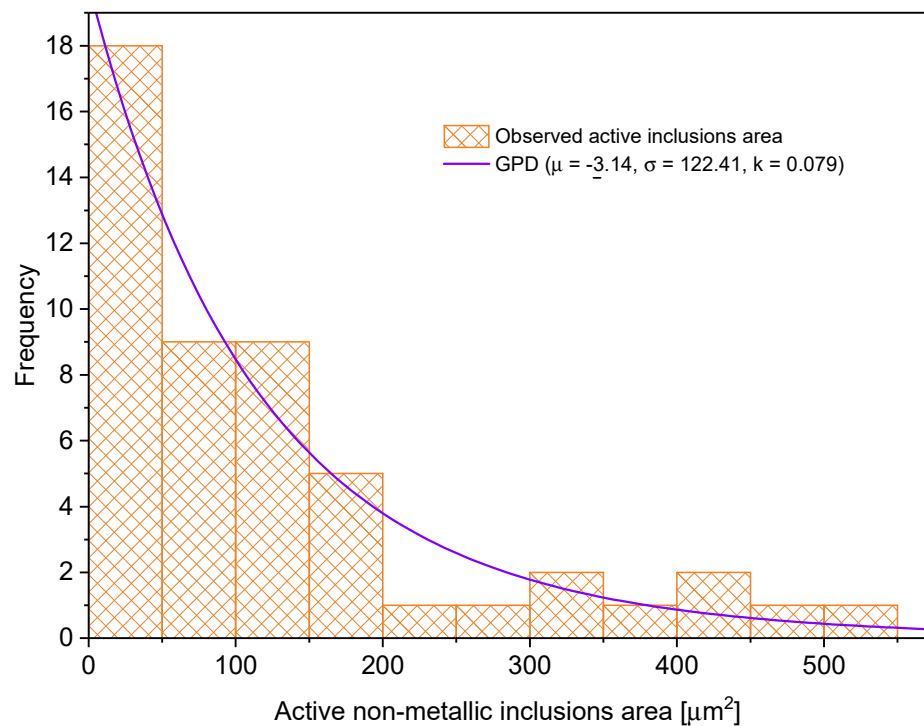


Figure 12. Histogram for active non-metallic inclusion area.

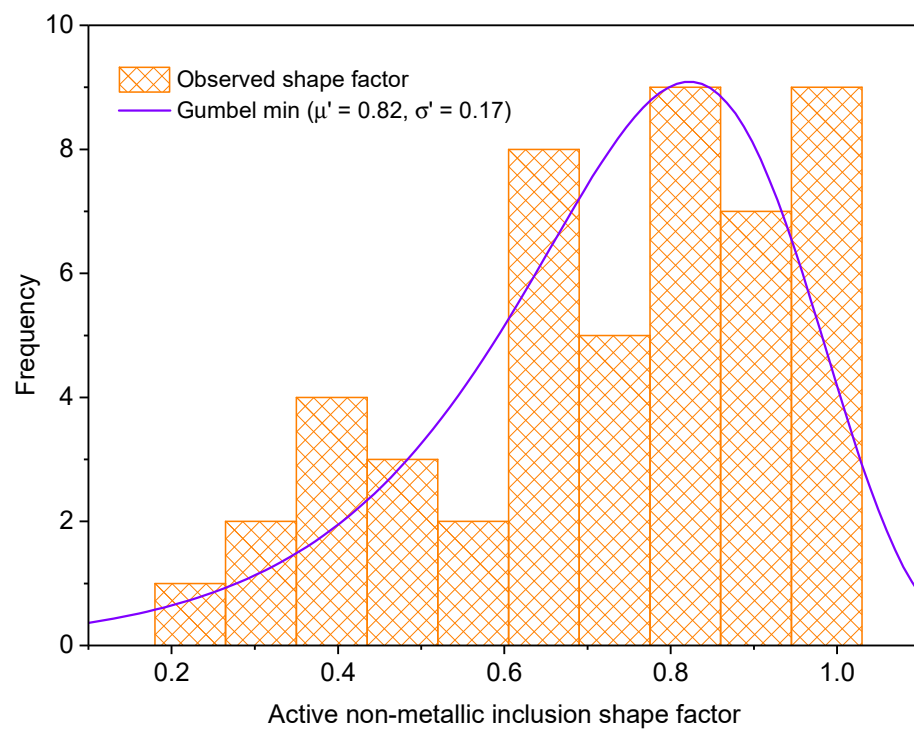


Figure 13. Histogram for active non-metallic shape factor.

For illustration purposes, as evidence that inclusions tend to generate defects, Figure 14 shows the appearance of a polished API 5L Grade B steel surface (see the inclusions in Figure 14a) and the same steel surface after being subjected to immersion tests (see the holes provoked by the pitting corrosion defects after the immersion test in Figure 14b).

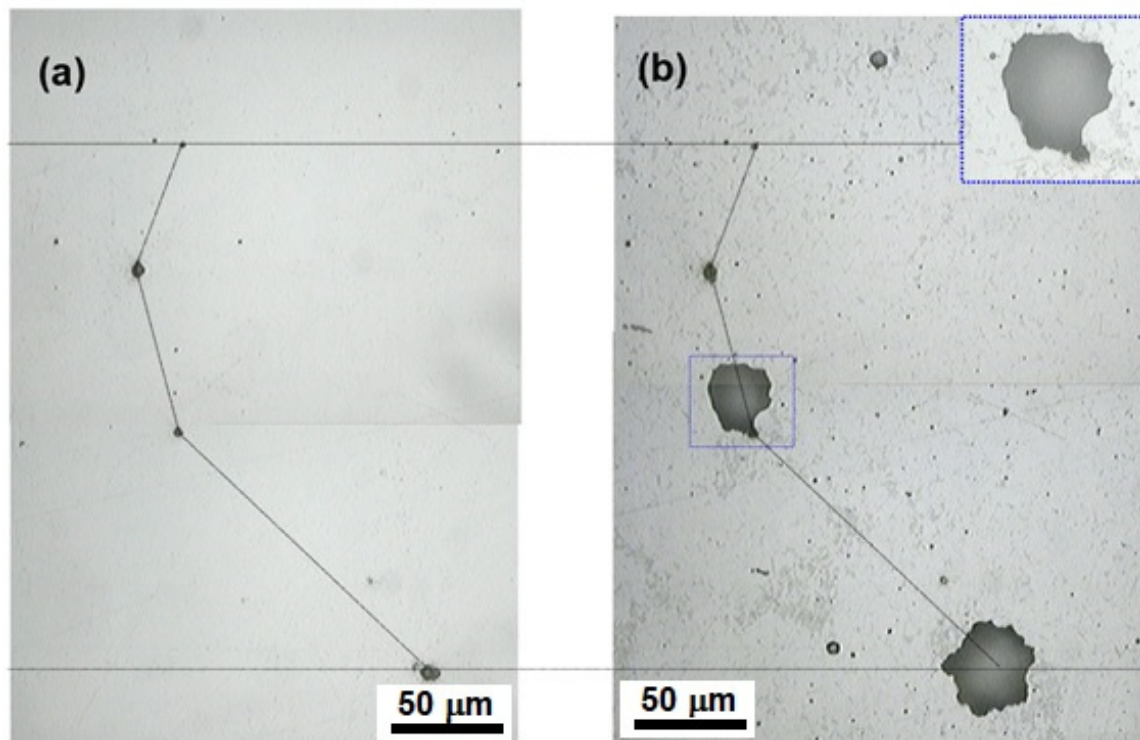


Figure 14. (a) API 5L X60 surface before immersion test and (b) after being immersed for 180 min.

Figure 15 shows micrographs of pits generated after the 30 min immersion test using different magnifications in a chemically etched steel surface, where inclusions with a shape factor close to one caused pit nucleation. Figure 15a–c do not allow to determine the shape factor correctly until we increase the magnification (see Figure 15d).

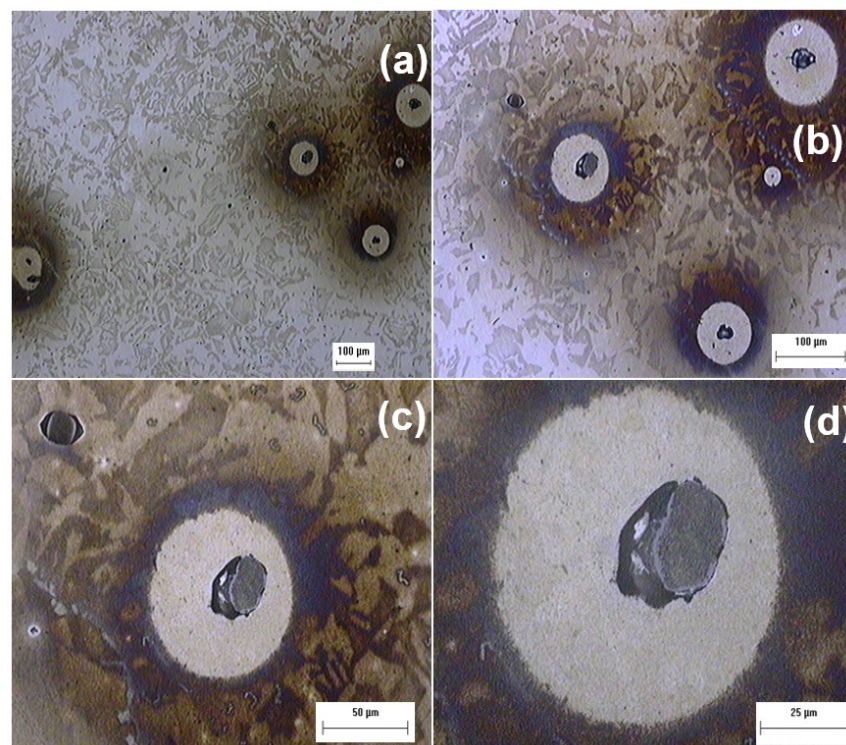


Figure 15. Micrographs of pits generated after inclusion dissolution at different magnifications.

One characteristic that is typically studied when oil and gas pipelines are in service is the pitting depth [10,12,38,39]. The main reason is that defects generated by localized corrosion threaten the hermeticity of pipes that transport hydrocarbons because they form stress concentrators that do not allow them to operate at the required pressure conditions [7,8]. For example, F. Caleyó and coworkers [39,40] determined using Monte Carlo simulations that localized corrosion defect depth exerts more influence in pipeline failure pressure estimation. For this reason, a histogram of the pitting depths generated due to inclusion dissolution (after 30, 60 and 180 min immersion tests) was plotted and adjusted to the GEVD (see Equation (6)), observing a high probability of accepting the null hypothesis (p -value greater than 0.05 in the K-S, A-D and chi-square tests). Figure 16 reflects this statement.

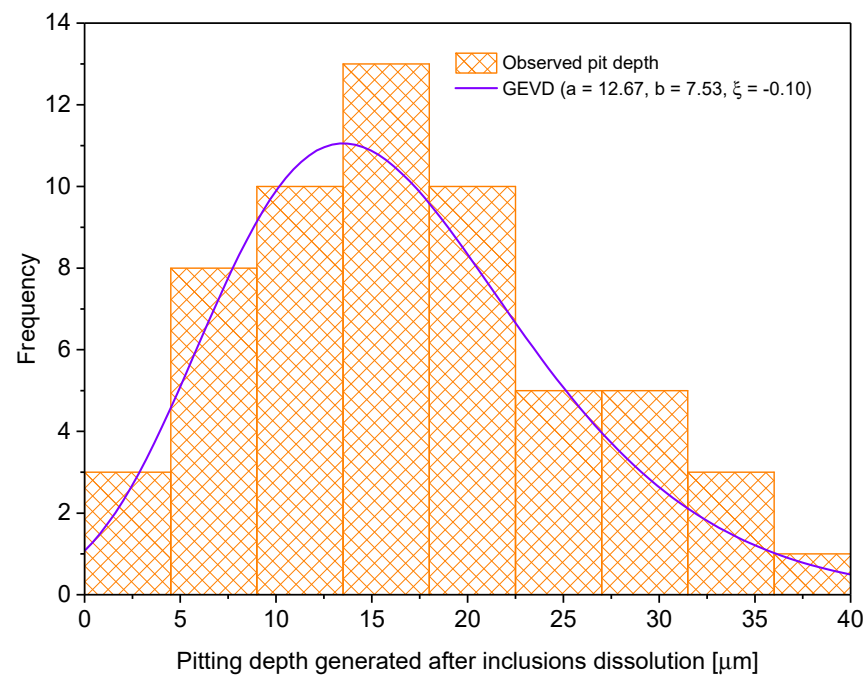


Figure 16. Pitting depth caused by inclusion dissolution.

3.3. Mid-Term Immersion Test

For the sake of analyzing the behavior of pitting corrosion depth evolution, the observed data were fitted to the GEVD because this distribution has been used previously to represent this evolution in the literature [40,41]. After fitting, it is feasible to observe the evolution of the shape parameter towards less negative values (see Table 4). In Figure 17, we notice that the pitting corrosion depth obtained after the 7-day immersion test shows a more positive kurtosis (of the majority of observed values close to the mean). On the other hand, the depths obtained after the 7-day immersion test generate a right-skewed distribution. As an example of the pits generated after the 1-day immersion test, Figure 18 shows the appearance of corrosion pits after the dissolution of active inclusions.

Table 4. GEVD parameters obtained after mid-term immersion tests.

Parameter	Duration of Immersion Test		
	1 Day	3 Days	7 Days
ξ	−0.29	−0.21	−0.19
b	5.18	5.94	8.55
a	15.66	16.70	19.86

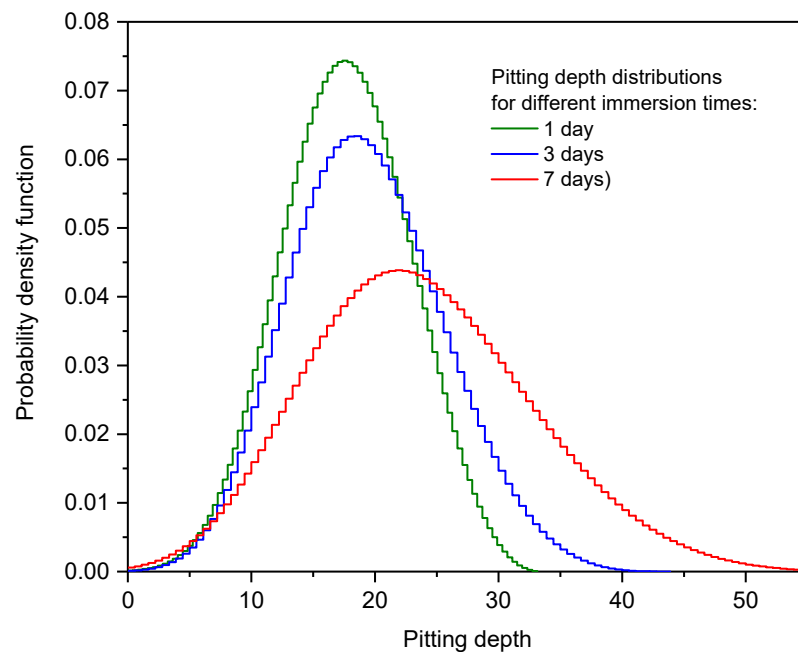


Figure 17. Pitting depth distribution after immersion tests of varying duration.

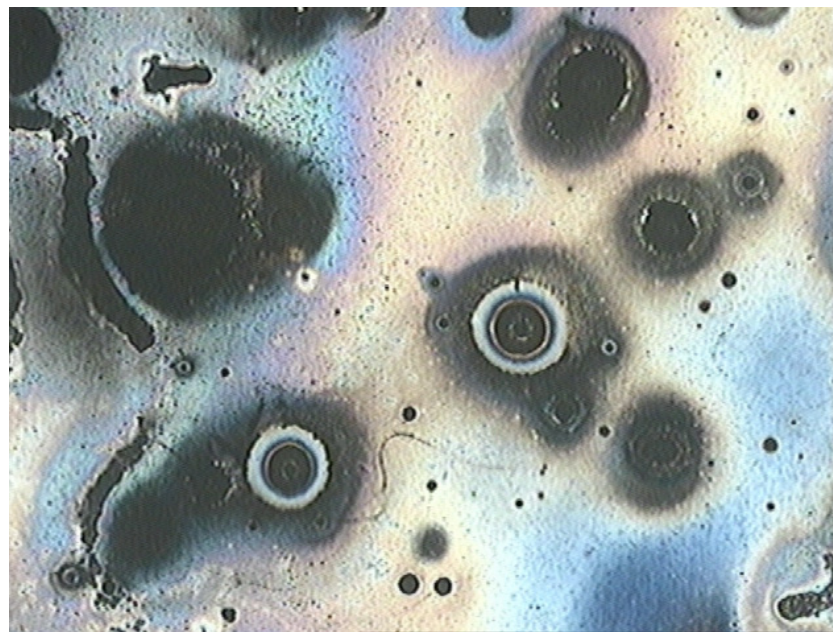


Figure 18. Pitting corrosion damage after 1-day immersion test.

3.4. Overall Analysis

The stochastic nature of pitting corrosion has been recognized since the 1950s [37,42,43]. It seems that this random characteristic could be caused by the same random nature of non-metallic inclusion features (e.g., length, width and shape factor) studied in the present research work. The Generalized Pareto distribution (GPD), Generalized Extreme Value distribution (GEVD) and Gumbel distribution are functions typically used to study the pitting corrosion phenomenon [37] and, in this investigation, remained as the preferred distributions that fit several pit characteristics (e.g., area and depth). The GEVD properly represents the evolution of pitting corrosion depth undergone after 1, 3 and 7 days of immersion.

Most of the inclusions found and analyzed were compounds of MnS, and they tended to become active inclusions when they came in contact with a corrosive environment. From the total number of inclusions found (regardless of their chemical composition), only about two to three percent of them became active. It seems that non-metallic inclusions with smaller areas tend to generate pitting corrosion defects with greater probability than bigger ones.

By observing Figures 15 and 18, we notice the darker aspect that can be attributed to a burn-like appearance due to the solution's acidity ($\text{pH} = 4.3$). This is evidence of the inclusion dissolution that motivates pit nucleation with an autocatalytic nature because the corrosion products and subproducts encourage further corrosion reactions, especially in acidic environments [42,43].

4. Conclusions

After considering the results obtained and their corresponding analysis, we can state the following conclusions to emphasize the relationship between non-metallic inclusions and pitting corrosion defects:

- Probability distributions such as those of Generalized Extreme Value, Generalized Pareto and Gumbel can satisfactorily represent the random nature of non-metallic inclusion characteristics, namely length, width and shape factor;
- The Generalized Extreme Value distribution fits adequately for pit depth histograms across mid-term immersion tests (1, 3 and 7 days). The shape parameter of this distribution tends to capture the change in the skewness and kurtosis because of the evolutions of the depth and the emergence of new and shallow pits;
- Inclusions with smaller areas tend to generate pits. In fact, there is a moderate correlation between inclusion area and pit area;
- MnS inclusions are more frequent in API 5L X60 steel than other type of inclusions. Likewise, MnS inclusions tend to be more active and prone to dissolution than others.

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