# The role of multi-phase metal content in corrosion and premature failure mitigation of steel equipment in oil refineries. Part 2

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The damage caused by crude oil impurities is in no way proportional to their small amounts, because there is an integrated system based on chemical composition and structure, chemical and electrochemical nature of corrosion processes, as well as sequence and synchronization between various damage mechanisms. Study of the influence of the heterogeneous metal content of the organic phase or steel matrix on premature failure of steel coils and equipment in oil refineries due to its aggressive role in corrosion processes allows us to reach certain rate-limiting stages of corrosion, which can help us describe better solutions to protect them against corrosion and premature failure. Industrial problems at which multiple sciences intersect represent challenging research tasks. On the other hand,

solving them is as difficult as it is, as much as it adds wider interconnection between the various sectors of science, which would provide a clearer and deeper understanding to the problems faced by researchers that contributes to a multidimensional scientific-industrial development.

*Key words:* crude oil, corrosion, steel coils, premature failure, inclusions, trace metals, nickel, vanadium, oil refinery, tubular furnace

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## Introduction

This part of the work is a continuation of our previous work [1]. It is well known that crude oil is subjected to heating process after desalting and dehydration in tubular furnaces prior to refining processes (distillation). Preheating destroys parts of the protective film or the scale of steel coils and thus initiates an endless cycle of corrosion in addition to other processes that eventually can lead to premature failure and associated accidents [2].

Inefficiency of desalting and dehydration processes of heavy crude oil in refineries leads to insufficient removal of many impurities and aggressive components, including, of course, metallic content, which, unfortunately, has not been given the enough attention that deserves, relative to its negative impact on various sectors [3-5].

There are a limited number of studies that have investigated the role of some metal cations, even simple ones, in the corrosion of steel in chloride containing media or others [6-10]. Anyway, they do not provide a general overview of the remaining cations, especially those with complex properties and overlapping effects, which requires much more research, especially on very complex media such as crude oil, that can provide a more complete understanding [11-15].

When a complex and multiphase mixture of hydrocarbon feedstock containing many aggressive components that have not been previously removed, and under the influence of high temperatures and pressures in microstructurally-defected

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steel coils and equipment. This will certainly lead to serious and irreversible deterioration of steel equipment, leading to its premature failure [15].

Premature failure is the inevitable result of multiple processes with mutually interacting effects that cumulatively affects steel structures. One of the most important of these processes is corrosion, which plays a very important role and occurs under different mechanisms and in various forms, which necessarily leads to lower quality (grade) of crude oil, and consequently to deterioration of oil products and fuels quality (grade), catalyst poisoning and premature failure of steel equipment, in addition to the high cost of repair, cleaning and replacement; as well as environmental pollution and its impact on humans. In addition to the costs of additional operations (traditional and non-traditional) for the upgrading of low-grade oil products and fuels [16, 17].

This article aims to take a further step in the same direction of our previous part by conducting comparative study in order to investigate the effect of trace metals of the organic (oil) phase on steel equipment most susceptible to corrosion, determining the rate-limiting stages of corrosion processes, and suggesting their possible mechanisms and kinetics.

## **Research methods**

After chemical pretreatment of crude oil with surfactants to remove organometallic trace elements, XRF-1800 X-ray spectrometry is used to compare the metal composition and content of the carbon residues, obtained from chemicallytreated crude oil combustion samples: LAB CENTER XRF-1800 / Sequential X-Ray Fluorescence Spectrometer (Manufacturer Shimadzu. Model XRF-1800, Japan).

Corrosion rates of steel coils used in tubular furnaces were determined through immersion testing by short-term laboratory simulation of real conditions [18]: either using water-oil emulsion with different organic trace metals contents to investigate the role of organometallics in the corrosion process; or using samples of heat-treated and untreated steel coils to study the role of intermetallic inclusions in the corrosion process.

## **Results and discussion**

From the terminology of crude oil dehydration and desalting, it is assumed that complete removal of mechanical impurities, salts and water should be performed, which, unfortunately, is far from being sufficiently and effectively achieved for several reasons. Such as: 1) the nature of the crude oil itself, which is becoming heavier because of the depletion of heavy oil reserves. 2) the storage and transportation conditions of crude oil. 3) the technical condition of storage tanks, transportation pipelines and steel processing equipment. 4) depending on the available quantity and quality of water used in the desalination process. Because of the large amount of oil produced, we need large quantities of water, which is assumed to have special characteristics for the desalination process. That in most cases is difficult to be achieved, and this is replaced by available water sources, which are water containing mainly large amounts of chlorides and sulfates of sodium, calcium, magnesium and other salts [2, 11, 19, 20].

The deterioration process depends on the microstructure (chemical, physical and mechanical properties) [21]. The heterogeneity of the steel microstructure in terms of the content of a large amount of inclusions of different types and phases, insufficient purification of the oil phase from heavy trace metals in organic phase (chelates or complexes), as well as ineffective dehydration and desalination processes - all this plays a very negative role, which is no less aggressive than the corrosive components themselves, because they can significantly accelerate localized corrosion [12, 22]. Thus, reducing of microstructure heterogeneity [23], preliminary purification of crude oil from trace metals [13] and more effective dehydration and desalting of crude oil very effectively slow down the corrosion rate [24].

Corrosion rate of steel equipment in oil refinery facilities can be slowed either by removing corrosive components and/or components that positively affect corrosion either by change the chemical composition of the crude oil (metal purification), or by changing the steel microstructure [25], through zone-leveling (controlling the distribution of impurities and inclusions) by special heat treatment without the need to add alloying elements during the steel making [24].

Study of the effect of trace metals of the organic phase of the crude oil on the corrosion rate of the steel coils and equipment:

In general, the trace metals in hydrocarbon raw materials can be divided into three main types: 1) Organometallic compounds with polar covalent bond; 2) Salts: in the forms of: 1-organic salts (carboxylates / naphthenates) 2-complex or chelates salts (complex ions), 3-inorganic salts (metals chlorides, sulphates,...); 3) Complexes or chelates with coordinate covalent bonds (dative bonds) between metals cations and organic ligands [14].

Due to the potential role of trace elements in organic oilphase compounds in various processes, including those that lead to the deterioration of the technical condition of steel equipment and its premature failure, it is extremely important to keep track of all their effects and roles.

The effect of the content of organometallic impurities (trace metals) was studied using different corrosive media with different trace metals content without changing the other conditions on steel coil samples (15X5M).

Taking into account that, vanadium and nickel complexes and even chelates make up a significant proportion of organometallic compounds in almost all types of crude oils. In our next study it is necessary to carry out an in-depth (detailed) research on extraction and tracing the behavior of vanadium and nickel in porphyrins, nonporphyrins and even asphaltenes through their purification (extraction) processes in order to give them sufficient attention due to their important and influential role not only in the oil refining industry, but also in environmental protection and human safety.

To study the influence of the presence of trace metals in the organic phase of crude oil on the corrosion of steel equipment, a chemical treatment with surfactants consisting of a mixture of several naphthenic acids (concentration of surfactants 3% at temperature 50 °C for 60 min) was carried out and the necessary analyses were conducted to measure vanadium and nickel contents. From our previous study [14], it was found that naphthenic acids and carboxylic (oleic) acids have different abilities to extract and bind organic metals in the form of naphthenates abd carboxylates [26], which are considered stronger surfactants (comparing to naphthenic and carboxylic acids), which in turn can be separated with the aqueous phase by centrifugation, as described in our previous study [13]. The obtained concentrate (containing extracted metals) can be converted to marketable products by traditional or non-traditional methods with the purpose of their utilization to make additional profits compensating the cost of preliminary processing [13].

If the metals with higher percentages in crude oil (vanadium and nickel) have a potential role in corrosion, this opens up great opportunities to study the role of each metal individually. Considering that vanadium and nickel are among the most common organometallic compounds and chelates with high molecular weights (porphyrins, non-porphyrins and asphaltenes), which tend to be present in greater quantities in heavy and dirty crude oils as compared to depleted light ones, it is therefore extremely important to track their possible behavior and role in corrosion processes to get a better and deeper understanding of corrosion mechanism of steel equipment in tubular furnaces, which ultimately leads to the reduction of industrial accidents, environmental disasters and economic losses [17].

Water-oil emulsions were formed from equal proportions of components (with two components in equal proportions),

initially using the crude oil phase (I0) with the aqueous phase. Then a mixture of various naphthenic and carboxylic acids was added and heated to 60°C for one hour followed by centrifugal separation. The oil phase centrifugally-separated from the first treatment (I), its treatment process with a new aqueous phase and a new mixture of acids was repeated three times in a row in the same way and under the same conditions: the second treatment - (II), the third - (III) and the fourth - (IV). The vanadium and nickel percentages in the four oil phases were then analyzed by burning certain weights of oil samples to produce carbonaceous residues, in which the nickel and vanadium contents were measured by XRF and then converted to percentages in the oil phases, as shown in the **Fig. 1**.

Vanadium and nickel concentrations were quantitatively converted to percentages to more comparatively determine the effect of retained (remaining) or removed metal content on the corrosion rate, as shown in **Fig. 2**.

As the number of times of chemical treatment with metals-extracting surfactants increases, the vanadium and nickel content continues to decrease in various proportions until the third stage, after which the decrease is not significant (Fig. 2). Therefore, three cycles of chemical treatment of the oil phase were sufficient for us ( $I_0$  - crude oil phase without treatment, I - crude oil phase after 1st treatment, II - after 2<sup>nd</sup> treatment, III - after 3rd treatment.

Quantitatively and for better comparison, we converted the corrosion rates units from mm/year to percentages (given that the corrosion rate of untreated crude oil is 100 %) and the same thing to the remaining and removed amounts of vanadium and nickel (given that the remaining is 100 % in crude oil and, in turn, 0 % removed in crude oil).

The high corrosion rate of the crude oil can be explained by the high concentrations of trace metals in the organic phase (Fig.2 -  $I_0$ ). Thus, higher percentages of some trace metals present in the organic phase can be released as cations and accelerate the corrosion process because of their electrochemical behavior and poisoning of the catalytic medium, especially from heaviest compounds closest to the aqueous layer on the bottom surface of the steel coil [27]. Although this rate-limiting stage is limited according to the values of formation constants for chelates and complexes, it is enough to deeply poison catalysts and dramatically accelerate corrosion due to the formation of new cathodic impurities that accelerate the reduction and therefore corrosion processes.

The removal of 62.27 % V and 65.33 % Ni from the oil phase after the 1st treatment reduced the corrosion rate by two times, and, in contrast, increased the corrosion resistance of steel equipment by 51.82 % compared to untreated crude oil. While the removal of 86.58 % vanadium and 90.04% nickel from the oil phase after the 2<sup>nd</sup> treatment reduced the corrosion rate to 25.51 %, i.e. about 3.9 times, and in return increased the corrosion resistance of the steel equipment to by 22.67 % by comparison to the oil phase after the first treatment. The remaining 11.35 % V and 5.72 % Ni in the oil phase after the 3rd treatment resulted in a limited decrease in corrosion rate from 25.51 % (II) to 19.43 % (III), i.e. by 1.3 times compared to the oil phase obtained from the second treatment, on the other hand, it led to a limited increase in corrosion resistance from 74.49 (II) to 80.57 (III), i.e. by 6.08 % compared to the oil phase obtained from the second treatment.

After the process of three-stage metals removing from the oil phase and before measuring the corrosion rates, the naphthenic and carboxylic acids that were used and remained in the oil phase were gradually removed through a three-stage washing using a new solution each time of sodium, calcium and magnesium salts, followed by a centrifugal separation of the oil phase, due to the emulsions formed [28]. Naphthenic and carboxylic acids can be recovered (extracted and reused

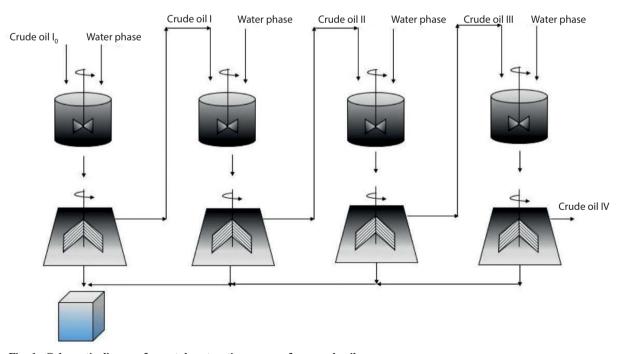


Fig. 1. Schematic diagram for metals extraction process from crude oil

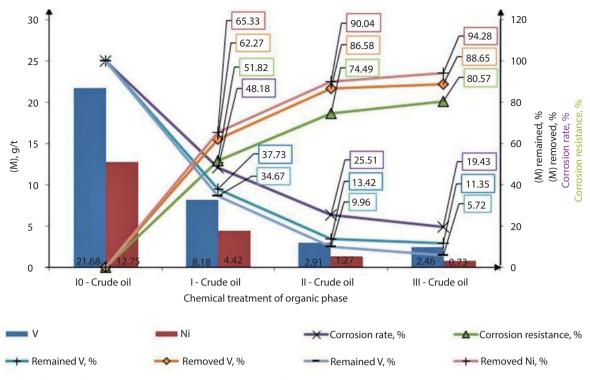


Fig. 2. Influence of V and Ni content on the corrosion rate of steel coils

again) by treating the centrifugally-separated aqueous phase after washing operations with hydrochloric acid, followed by separating the organic from the aqueous phases using a separating funnel [13, 14].

#### Mechanism of the premature failure of the steel coils

The premature failure mechanism of steel coils can be explained by the following sequential steps, as shown in the **Fig. 3**, where «IoP - in organic phase, «OP - organic phase, «Inc - inclusions.

It should be noted that the black layer in the figure 3 indicates the protective-passive layer as in the case of stainless steel coils and / or scale obtained by various processes in the case of general (uniform) corrosion of low-carbon steel coils (corrosion products, deposits of inverse solubility salts and coking). Sulfide inclusions are presented at the top, and sulfide-oxide inclusions are presented at the bottom in the Fig. 3.

Partial destruction and falling-off of parts of the protective layer or scale and the appearance of contact surfaces (interfaces) (boundaries) between the protective layer or scale with inclusions (sulfides) is the initial point of localized corrosion due to the contact surface of the protective layer or scale / sulfide inclusions being more or less electrochemically-active than the adjacent (surrounding) steel areas. There are micro-voids between the inclusions and the surrounding steel structure, which are formed due to their different response to heating and cooling processes during steelmaking processes as a result of their different thermal expansion coefficients [24, 29].

A temporary galvanic pair (micro-electrochemical pair) is formed in which the contact surfaces of the inclusions with the protective layer or the scale are anodic areas compared to the cathodic areas of the protective layer or the scale. This occurs when the sulfide inclusion is less electrochemicallyactive (more noble) than the surrounding steel, then the surrounding steel is the preferred anodic area. Thus, the surrounding steel area around the inclusion starts corroding, and the flowing medium can penetrate through the microvoids around the inclusions, reaching new contact surfaces between the inclusions and the steel structure. The inclusions act as an electrically conductive bridge, transferring electrons from anodic to cathodic areas to be consumed in reduction (depolarization) reactions, because the protective layer or scale is more noble than the sulfide inclusion. Then a permanent galvanic pair is formed in which the steel structure is the anodic area and the protective layer or the scale is the cathodic area. As a result of the increased local acidity due to steel corrosion and hydrolysis of iron cations, the sulfide inclusion starts dissolving chemically.

But when the intermetallic sulfide impurities are more electrochemically-active (less noble) than the surrounding steel structure, a temporary galvanic pair is formed in which the intermetallic sulfide impurities are the anodic area and the protective layer or scale is the cathodic area. The inclusion then begins to electrochemically dissolve (corrode) and micro-voids are formed, and the steel transfers electrons to the protective layer or scale for consumption through cathodic reactions (depolarization) because the protective layer is more noble than the steel.

The iron cations obtained from the corrosion electrically attract the chloride anions to provide electro-neutrality within the formed pits, which weakens the mechanical stability of the protective layer or the scale and the inclusions edges because of the adsorption that occurs.

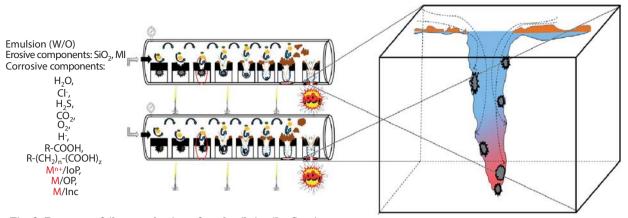


Fig. 3. Premature failure mechanism of steel coils in oil refineries

In parallel, the iron cations produced by the corrosion are hydrolyzed, releasing hydrogen ions, which, together with the chloride ions, form strong electrolyte HCl, fullydissociated, which leads to a local decrease in pH within the pit. Decrease of pH forces other inclusions to be dissolved, releasing their own metal cations, which in turn can be hydrolyzed, releasing additional hydrogen ions, in addition to other highly aggressive products (NH<sub>3</sub>, H<sub>2</sub>S, silicic acid H<sub>4</sub>SiO<sub>4</sub>,...), which contributes to the acceleration of localized corrosion and corrosion under solid deposits, since ammonia can combine with HCl to form the aggressive ammonium chloride NH<sub>4</sub>Cl [30, 31].

Electrochemical corrosion, caused by the formation of galvanic pairs, is concurrently intensified in parallel by the formation of concentration cells. Steel coils eventually deteriorate due to a vicious cycle of overheating of the most deeply-corroded areas and localized corrosion that aggressively and severely accelerate each other, resulting in rupture, and thus premature failure [32], as shown in **Fig. 4**.

In advanced stages of deep localized corrosion of the steel surface in contact with the hydrocarbon flow medium, i.e. when pits or cracks approach the outer surface of the steel coil, this leads to increased rates of overheating, which in turn leads to the decomposition of corrosion products and other hydroxides into oxides and water (water hydrolysis recovery stage), providing an additional source of water which dramatically accelerates corrosion. Exactly the same thing happens in corrosion under the protective thermal insulating layer due to high temperatures, except that the water hydrolysis recovery stage contributes (promotes) corrosion processes from the early stages of corrosion.

## Kinetic study of the premature failure of the steel coils

Localized corrosion, either on the inner surface of the steel coil in contact with the flowing medium or on the outer surface of the steel coil under the thermal insulation layer, is initiated, unlike uniform corrosion, from areas of steel around sulfide inclusions from an energetic or electrochemical perspective, depending on the type of inclusion and its electrochemical behavior [33, 34]. This occurs for two reasons, regardless of whether these inclusions are more or less electrochemically-active than the surrounding steel: first, because these areas are microstructural defects and are considered to be the least energetically stable, and therefore have a stronger tendency to transform into the most energetically-stable forms, and second, because there are micro voids around the inclusions (due to their formation conditions during various heat treatments associated with the steel melting stages), and, therefore, the flowing medium, in particular the refreshed (regenerated) aqueous phase microlayers, can reach these areas of steel to initiate corrosion.

During electrochemical dissolution (corrosion) or chemical dissolution (due to pH values) and under the influence of density, the sulfide inclusion moves upward after being released from the steel cage in the corroded pocket, promoting conduction of electrons to the cathodic areas (scale or protective layer) for their consumption due to the oxygen reduction reaction (depolarization). This leads to a change in the nature of the protective layer or scale, which becomes more capable of ionic and electrical conductivity as the corrosion intensifies, resulting in an increase in its thickness. However, on the other hand, corrosion under solid deposits increases the potential for some of their parts to fall-off along with the flowing medium.

Thus, localized corrosion is much more serious, dangerous and tricky than general (uniform) corrosion because around-inclusionary corrosion (corrosion around solid inclusions) grows and spreads vertically deep into the steel structure compared to corrosion under solid deposits, which grows and spreads horizontally. This is in addition to the fact that the cross-sectional area of the corroded steel surface in the general corrosion is much larger than in local corrosion, making the anodic current density in local corrosion much larger than in general corrosion.

Since the dissolution of gases (oxygen) in water decreases with increasing temperature, one should expect that the ratelimiting stage should be the dissolution of oxygen in water. However, in both cases of corrosion (on the inner surface of the steel coil or on the outer surface under the protective thermal insulation layer) we have a closed system. In this system, the remaining water in the oil phase is constantly supplied, and the condensed moisture or water under thermal insulating layer is already trapped and/or which penetrated through the defective points of the thermal insulation layer. This, in turn, retains dissolved oxygen (neither releasing nor evolving it) in both cases and at the same time maintains con-

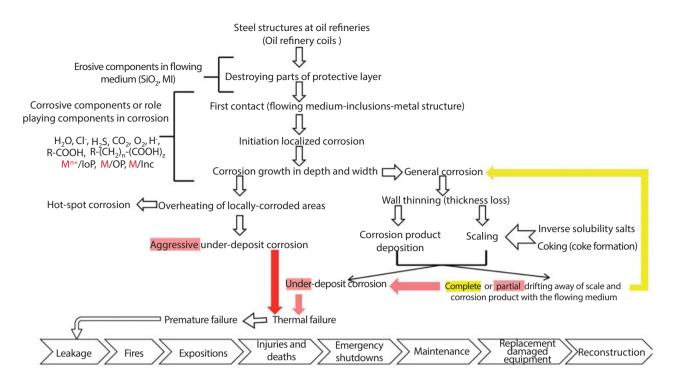


Fig. 4. Schematic diagram of the initiation and growth of corrosion leading to premature failure of steel coil at oil refineries

stant contact with the steel surface, which ensures electron consumption (depolarization) during reduction reactions and thereby ensures continuous corrosion.

From our studies (parts 1 and 2) we can conclude that there are three rate-limiting stages of determining (controlling) the corrosion rate of steel coils or equipment at oil refineries: 1st rate-determining stage: diffusion of the dissolved oxygen molecules to the cathodic areas; 2<sup>nd</sup> ratedetermining stage: electron transfer (electronic conduction) from corroded to cathodic areas; 3<sup>rd</sup> rate-determining stage: release of heavy trace metals cations from heavy organometallic compounds, the closest to the micro-layer of the aqueous phase on the inner surface of the steel coils.

Convection of micro-layers of the aqueous phase on the lower surface of steel coils (due to mutual dependence of densities and heating temperatures) and the continuous feeding (supply) of oxygen with hydrocarbon raw materials, besides the role of some formed intermediate compounds or complexes of trace metals with high affinity for oxygen (e.g., vanadium intermediates) on the cathodic areas are responsible for weakening the 1st rate-determining step, which increases the accessibility of dissolved oxygen to reach cathodic areas [35, 36].

The inclusions that are released from the steel cage during the initiation and growth of corrosion are responsible for the weakening of the 2<sup>nd</sup> rate-determining stage, due to their densities and ability to transfer electrons from anodic to the cathodic areas.

Increasing the concentration of  $Fe^{2+}$  iron cations above a certain threshold due to high corrosion rates, especially in the advanced stages of corrosion, can lead to partial dissociation of some chelates (even in a very small percentage) according to the Le Chatelier principle, and thus can limitedly enhance electro-

philic exchange (between trace metal cations and Fe<sup>2+</sup>) due to the values of the chelate formation constants, but sufficiently enough to form micro-cathodic impurities that accelerate the electron consumption through reduction reactions and hence the corrosion rate. In other words, iron cations can compete with a small fraction of the transition heavy metal cations in chelates for binding to organic ligands due to an increase in their concentration, resulting in a shift in the chelate equilibrium of trace elements toward its limited dissociation, while the chelate equilibrium of Fe<sup>2+</sup> toward its limited formation. This increase in Fe<sup>2+</sup> concentrations is responsible for the weakening of the 3rd rate-determining stage. This is in addition to the role of the recovered hydrolysis water in the advanced stages of corrosion of the inner surface of the steel coils in contact with the flowing hydroacryon (and from the initial stages of corrosion of the outer surface under the thermal insulation layer), which accelerates corrosion at dramatic rates.

## **Conclusions and recommendation**

A comparative experimental study proved the role of trace metals in high-molecular-weight organometallic compounds of crude oil, such as chelates and complexes (asphaltenes and porphyrins), in accelerating corrosion processes and premature failure of steel coils and equipment.

The study confirmed that the cause of the premature failure of steel coil was mainly due to thermally-accelerated aggressive localized corrosion, which is intensified (enhanced) by the continuous supply of dissolved oxygen during hydrocarbon raw materials feeding, in addition to the role of recovered hydrolysis water in the corrosion processes, apart from of the effects of overheating. The corrosion resistance of steel coils and equipment can be indirectly improved by purifying crude oil from impurities of trace metals due to their cross-interacting influences with other aggressive components under mutually reinforcing (stimulating) mechanisms.

The corrosion process is controlled through three ratedetermining stages: the first is the diffusion of dissolved oxygen molecules to the cathodic areas, the second is the electronic transfer from the anodic to cathodic areas, and the third is the release of heavy trace metals cations from heavy organometallic compounds. Despite the presence of these rate-limiting stages, the corrosion rate is constantly increasing due to the various factors that mitigate (weaken) the limitations to these stages.

There is a need for further research in which the electrochemical aspect is discussed in more detail. Our next articles will discuss in detail the methods, mechanisms and kinetics needed to purify the oil phase from trace metal impurities because of their negative role in both the safety of refining processes and environmental pollution.

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