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Failure analysis in heat exchanger tubes from the top system of the regeneration tower of the hydrotreatment unit in an oil refinery: a case study

Análise de falha em tubos de trocador de calor do sistema de topo da torre regeneradora da unidade de hidrotratamento em uma refinaria de petróleo: um estudo de caso

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ABSTRACT

This paper presents a failure analysis performed on tubes of a heat exchanger. Heat exchangers are among the most common equipment in an oil refining industry, as heat transfer is one of the most important and common processes in chemical and petrochemical industries. The equipment analyzed was the condenser of the top system of the diethanolamine regeneration tower of the naphtha hydrotreating unit of an oil refinery. The tubular bundle consists of 78 tubes in the ASTM A213 Grade 316L specification, with 19.05 mm in diameter and 1.65 mm in wall thickness. The tubes are subjected to the aqueous flow of H_2S and diethanolamine on the outside and cooling water on the inside. After a scheduled general shutdown in July 2011, after six years of continuous operation, the loss of thickness in several tubes was observed. The result of the failure analysis indicated that, through visual inspection, the corrosive process occurred from outside to inside the tubes. The eddy current test indicated an active corrosive process with a corrosion rate of 0.16775 mm / year. The ultrasonic inspection performed through the internal rotating inspection system (IRIS) confirmed the loss of thickness and the presence of perforated tubes, with consequent loss of containment. Amine corrosion in the presence of H_2S was identified as the basic failure mechanism, aggravated by DEA concentration higher than 20 wt.% in the analyzed equipment. In October 2019, the tubes of this exchanger were replaced and the proposed solution to mitigate the problem was to dilute the solution with washing water, to obtain amine solution concentrations below 5% by mass in the beam tubes of the heat exchanger.

Keywords: Failure analysis; Oil refinery; Heat exchanger; Hydrotreatment; Dietanolamine regeneration tower.

RESUMO

Este artigo apresenta uma análise de falhas realizada em tubos de um trocador de calor. Trocadores de calor estão entre os equipamentos mais presentes em uma indústria de refino de petróleo, pois a transferência de calor é um dos processos mais importantes e comuns em indústrias químicas e petroquímicas. O equipamento analisado foi o condensador do sistema de topo da torre de regeneração de dietanolamina (DEA) da unidade de hidrotratamento de nafta de uma refinaria de petróleo. O feixe tubular é constituído por 78 tubos na especificação ASTM A213 Grau 316L com 19,05 mm de diâmetro e 1,65 mm de espessura de parede. Os tubos são submetidos ao fluxo aquoso de H_2S e dietanolamina na parte externa e água de resfriamento na parte in-

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terna. Após uma parada geral programada ocorrida em julho de 2011, após seis anos de operação contínua, foi observada a perda de espessura em vários tubos. O resultado da análise de falha indicou que, através da inspeção visual o processo corrosivo ocorreu de fora para dentro dos tubos. O ensaio de correntes parasitas indicou um processo corrosivo ativo com uma taxa de corrosão de 0,16775 mm/ano. A inspeção ultrassônica realizada através do sistema de inspeção rotativa interna (IRIS) confirmou a perda de espessura e a presença de tubos perfurados, com consequente perda de contenção. A corrosão por amina em presença de H_2S foi identificada como o mecanismo básico da falha, agravada pela concentração de DEA superior a 20% em massa. Em outubro de 2019, os tubos deste trocador foram substituídos e a solução proposta para a mitigação do problema foi a diluição da solução com água de lavagem, para a obtenção de concentrações da solução de amina abaixo de 5% em massa nos tubos do feixe deste trocador de calor.

Palavras-chave: Análise de falhas; Refinaria de petróleo; Trocador de calor; Hidrotratamento; Torre de regeneração de dietanolamina.

1. INTRODUCTION

Heat exchangers are among the most common equipment in an oil refining industry, as heat transfer is one of the most important and common processes in chemical and petrochemical industries [1]. Heat exchangers are generally found in tubular and plate forms. The material selection for this type of equipment involves several important considerations, such as corrosion resistance, thermal conductivity, mechanical resistance, weight**,** and cost. Perhaps the biggest challenge today is the achievement of high reliability in the service due to exposure to aggressive environments in a wide range of operational parameters [1]. The most used exchanger in the processing industries is the hull-tube type, with one of the fluids passing through the tubes, and the other through the space between the hull and tubes.

In the oil industry, heat exchangers are commonly used to oil heating or gas cooling. In the case of oil, the main purpose of its heating is to facilitate the removal of impurities, as this temperature increase promotes better gas separation in high viscosity oil, in addition to improving the separation of water in oil [2].

Hydrotreatment is an oil refining process that consists of the insertion of hydrogen gas, under appropriate pressure and temperature, in the currents derived from the cuts of the oil distillation. To meet market specifications, environmental legislation, in general, gasoline and diesel undergo the hydrotreatment process, which aims to remove contaminants, especially sulfur and its compounds, and stabilize the fuel through catalytic hydrogenation reactions [3, 4]. The treatment of diethanolamine is a specific process used to remove hydrogen sulfide from gas fractions, consisting of the use of solutions with ethanolamine's (mono, di and tri) that have the property of combining with hydrogen sulfide, through selective solubilization, forming stable products at temperatures close to the environment [5]. The products formed, when submitted to heating, decompose, regenerating the original solution and releasing previously absorbed gases, which can then be sent to the sulfur recovery units of oil refinery.

Amines, such as monoethanolamine in aqueous media, are widely used to remove carbon dioxide in gas flows [6]. Monoethanolamine is the most widely used amine, but it is currently being replaced by diethanolamine, with greater regenerative power and low cost, which allows gains in performance and initial absorption [7, 8]. There are reports that indicate the use of amine mixtures for specific applications [9]. Generally, such processes for removing contaminants by amines occur at room pressures and temperatures [7-9]. The peak of the absorption by the amines occurs around 20 wt. % to 40 wt. % of the amine concentration [7-9]. Diethanolamine is an organic chemical compound, which is both a secondary amine and an alcohol and has the following molecular form: $C_4H_{11}NO_2$. The diethanolamine acts as a weak base. There are reports of several applications of amines for absorption, such as in oil refineries to remove H_2S in acid water units and stripper columns [10]. In petrochemical industries, amines are used to absorb ethylene oxides. In general, applications involving oil processing are applied to amines respectively in the removal or extraction of carbon dioxide and hydrogen sulfide gas. In amines, in general, alkanolamines have at least one hydroxyl group and an amine group in their chemical structure. Hydroxyl groups increase solubility in water and reduce the vapor pressure of alkanolamines. The amine group in aqueous solutions provides the necessary alkalinity for the acid gas absorption process. DEA solutions without acidic gases induce smaller corrosion rates than water [11]. Lean amine solutions are generally not corrosive because they have either low conductivity and or high pH, but an excessive accumulation of heat stable amine salts above 2% can generate corrosion processes [12]. Figure 1 shows the structural formula of the diethanolamine most used in the process of removing acid gases.

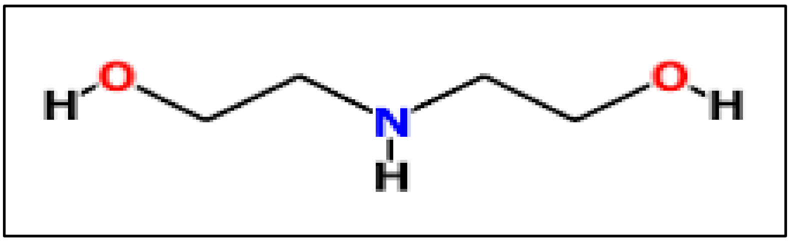

Figure 1: Chemical structure of the diethanolamine.

During the acid gas removal process, H_2S reacts with the hydroxyl group, being an extremely fast reaction. In general, the amine absorption process occurs according to the equilibrium reactions [11]:

$$\text{Amine} + H_2S = \text{Amine } H^+ + HS^- \quad (1)$$

The general dehydrogenation reaction of diethanolamine is endothermic (estimated at 12 Kcal/mol of DEA) and provides reasonable reaction rates and selectivity at temperatures between 140 ºC and 200 ºC [13]. Figure 2 shows the heat exchanger of the top system of the regenerating tower.

Figure 2: Heat exchanger of the top system of the DEA regenerating tower.

Figure 3 shows the regeneration DEA tower. The operating pressure is 1.2 Kg/cm², at 177ºC, capacity of 10.4 m³, length of 15,200 mm, material of ASTM SA-264 (structure in carbon steel ASTM SA-516 Gr. 70 + internal cladding in austenitic stainless steel ASTM SA-240, 317L).

Figure 3: The DEA regenerating tower in the oil refinery.

Failures in heat exchangers occur due to defects introduced into pipes during the stages of manufacturing, handling, testing, shipment, and storage or during start-up, shutdown, and normal operations of the heat exchanger [14]. A variety of non-destructive tests can be used to detect failures in heat exchangers such as a simple visual inspection of their components and the application of monitoring techniques, such as Eddy current testing, hydrostatic testing, penetrating liquid testing, testing by magnetic particles, industrial radiography, thermography, ultrasonic tests and metallographic analysis [1].

The most common modes of failure in heat exchangers include fatigue, creep, corrosion, oxidation, and hydrogen attack. The causes of failure include scale, salt deposition, weld defects and vibrations. The inadequate selection of material and operation in abnormal conditions influence the occurrence of these phenomena, causing damage to the hull and the tubes of the heat exchangers [15]. These damages are externalized through the occurrence of cracks, leaks, thickness loss, weakening of the material, with consequent limitation of operation of the production units, loss of operational life and possibly occurrence of accidents with economic and production loss, damage to the environment and loss of life.

In general, amine corrosion refers to generalized or localized corrosion that occurs mainly in carbon steel in an amine treatment unit; this corrosion type is not caused directly by the amine itself, but by the dissolution of acid gases such as carbonic acid and H_2S as degradation products. This corrosion type depends on the amine concentration, contaminants, temperature, and flow rate. The corrosion morphology is usually uniform, but at high flow rates or in the presence of turbulence, the corrosion may be localized. In carbon steels, this flow rate is limited to about 0.92 to 1.82 m/s in a rich amine solution and 6.09 m/s in a poor amine solution. Various gaseous flows of chemical processes discharged into the atmosphere or used as fuels must be cleaned beforehand, to remove contaminants such as carbon dioxide, hydrogen sulfide and carbonyl sulfide, the removal of these acid gases is extremely necessary in the oil refining industry, which can cause damage to equipment due to corrosion [16]. Besides that, the degradation of the DEA and the formation of heat-stable salts (HSS) lead to severe corrosion problems [11]. In addition, corrosion problems of steels exposed to solutions of DEA and H_2S can be aggravated by the presence of oxygen [11]. GARCIA-ARRIAGA et *al.* [11] concluded that the presence of oxygen in a sweetening plant should be avoided as DEA degradation can be produced with the subsequent decrease in chelating process efficiency and the increase in corrosion problems.

The most used non-destructive testing (NDT) for monitoring corrosion in stainless steel heat exchangers is the Eddy current testing. This NDT method is sensitive to localized corrosion, general corrosion, and cracks in internal and external tube surfaces [17]. The technique can be used to control the phases of production, final product or to detect product discontinuities in service, such as fatigue cracks or changes in the mechanical or physical properties of the part material. The IRIS (Internal Rotation Inspection System) is a non-destructive ultrasound testing technique. The IRIS test, using the conventional ultrasonic pulse-echo principle, allows the measurement of thicknesses, identification of pits, abrasion, and cracks in small diameter tubes (tubes of heat exchangers and boilers, pipes in chemical, petrochemical and nuclear energy industries). The probe produces very precise and detailed results and can be used safely in studies of integrity assessment and calculations of remaining useful life [18].

This work presents a failure analysis performed on tubes of a condenser of the top system of the diethanolamine regenerating tower of the oil cracked naphtha hydrotreating unit. The tubes are subjected to the aqueous flow of H_2S and diethanolamine in the external region and cooling water in the internal region.

2. MATERIALS AND METHODS

The data analyzed were obtained from maintenance and inspection files of the inspection management of equipment and facilities at an oil refinery during maintenance shutdown activities. Figure 4 shows the tube bundle analyzed. The initial failure analysis of the tubes was performed by using external and internal inspection. Chemical analysis of tubes was performed by using X-ray spectroscopy, using the Niton Analyzers alloy analyzer, model XL3t-800 Thermo Scientific.

Figure 4: Tubular bundle analyzed.

The analysis was based on the investigation of a sample through interactions between particles or electromagnetic radiation and matter, analyzing the X-rays emitted by matter in response to the incidence of charged particles. Eddy current test was performed by using the Olympus Nortec 600 equipment with tube probes with a 316L steel test standard.

Ultrasonic thickness measurement was performed using Internal Rotation Inspection (IRIS) made with 05 PAT equipment, 14 mm diameter centralizer, standard 316L steel tube, normal type transducer, test tube probe, 12 mm diameter turbine, 5.790 m/s sonic speed, 24 dB primary gain, 45° turbine mirror, 15 MHz transducer frequency, and coupling in demineralized water (Figure 5).

Figure 5: Internal Rotation Inspection (IRIS) equipment test.

The IRIS (Internal Rotation Inspection System) is a non-destructive ultrasound testing technique that consists of a probe inserted in a tube flooded with water. As the probe moves, data is displayed and recorded. All measurements made during the complete circumferential scan of the tube are shown on the computer screen, producing images in real time, allowing the mapping of detected non-conformities. Figure 6 shows the schematic drawing of the probes which transmit an ultrasonic pulse, reflected at right angles by the rotating mirror (45º) housed in the probe turbine assembly.

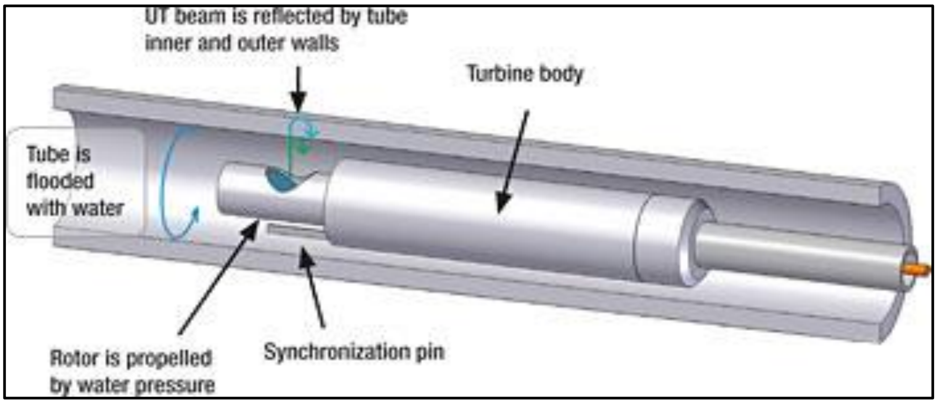

Figure 6: Probe turbine assembly.

After the initial visual inspection, the tubes located at the upper side of the heat exchanger were taken for internal and metallographic analysis. Samples were extracted from the tubular bundle that had 78 tubes, with a diameter of 19.05 mm, wall thickness of 1.65 mm and a total length of 6,096 mm. These samples were cut to a length of 300 mm and sectioned longitudinally. Metallographic analyzes were performed by inlaying in acrylic resin and polishing with sandpapers up to 1000 mesh. The final polishing was performed with 5 micron and 10 micron diamond paste and distilled water was used for final cleaning and steel samples were dried with heated air. The reagent used in metallographic analysis was oxalic acid solution at a concentration of 10 wt. %. A Unimet Union 9000 measuring microscope equipment was used.

3. RESULTS AND DISCUSSION

3.1 Chemical composition

The chemical composition of failed tube was compared with the specification of ASTM 316L stainless steel in Table 1.

Table 1: Chemical composition of tubes (wt. %)

SAMPLE	C	Mn	Cr	Ni	Mo
Failed tube	-	1.76	16.81	10.95	2.05
Specification ASTM A-213 Grade 316L	0.03	2.00	18.00	14.00	3.00

The measured levels are within the specification ranges of the ASTM A213 Grade 316L standard. The carbon content was not measured.

3.2 Visual Inspection

Figure 7 shows the external surface of the tubes after cleaning and cutting in the longitudinal section (half-cane). An extensive area of corrosion is observed on the outer sides of the beam tubes, the contact area of the metallic material and the fluid of the hull (H_2S + DEA).

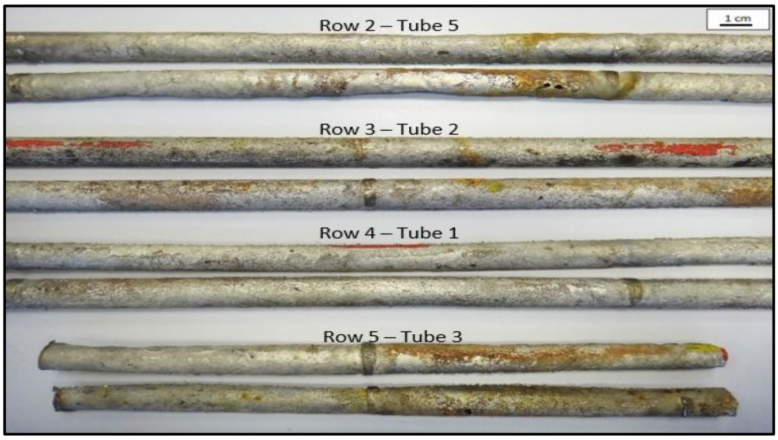

Figure 7: External visual inspection of the heat exchanger tubes.

Figure 8 shows the inner surface of the tubes after cleaning and cutting in the longitudinal section of the tubes (half-cane). Visually, the inner surface of the tubes is unchanged, without evidence of a corrosive process, the fluid inside the tubes is cooling water. Therefore, it is proved that the corrosive process in these tubes occurred from the outer surface to the inner surface.

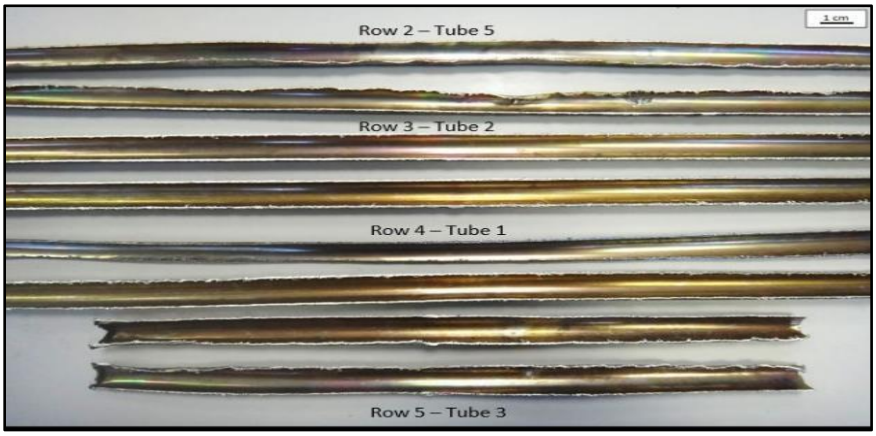

Figure 8: Internal visual inspection of the heat exchanger tubes.

Figure 9 shows a detail of a hole found in one of the tubes in the upper pass region of the heat exchanger. This is the tube located in row 2 and column 4, detected by the IRIS ultrasonic test.

Figure 9: Detail of tube punctured in the heat exchanger beam.

3.3 Eddy current test

The exchanger's inspection history reveals that during the first programmed general stop in September 2017, the tubular beam was submitted to non-destructive tests by Eddy currents, which, by means of microstructural variations with different magnetic properties, and, consequently, different impedances, allowed the detection of cracks and holes in tubes. Figure 10 shows the spatial distribution of tubes tested by Eddy currents.

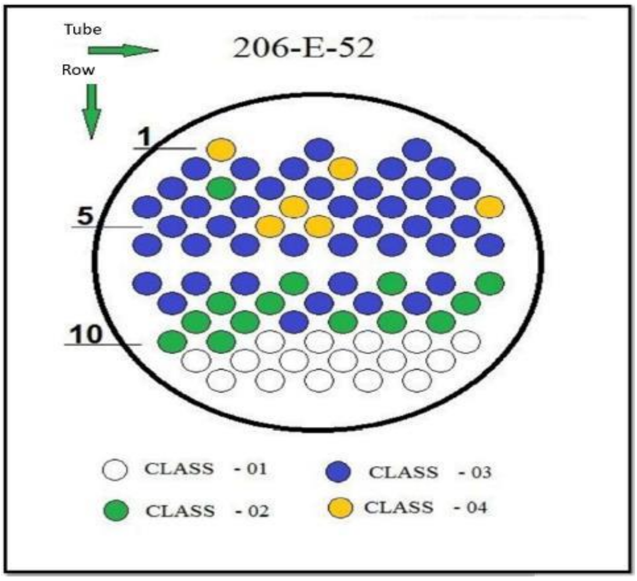

Figure 10: Spatial distribution of tubes tested by eddy currents.

The results of Eddy current test performed on the investigated heat exchanger tubes are described below in Table 2.

Table 2: The results of Eddy current test performed on the tubular bundle.

CLASS	THICKNESS LOSS	NUMBER OF TUBES
1	0% to 20%	16
2	21% to 40%	14
3	41% to 60%	42
4	61% to 80%	6
Total of tubes		78

The definition of inspection class depends directly on the thickness loss. The four inspection classes were defined in the test. As observed about 53% of the tubes (42 out of a total of 78) presented a thickness loss between 41% and 60% in September 2017, with the nominal diameter of the tube being 19.05 mm and wall thickness of 1.65 mm when installing and starting the heat exchanger (July 2011).

The smallest residual thickness was 0.45 mm; thus, a corrosion loss of 1.20 mm was identified in thickness. In this way, the tubular beam of this heat exchanger was classified with a remaining useful life of one-year operation, considering a corrosion rate calculated for this situation of 0.1677 mm/year. The six tubes showed a thickness loss between 61% to 80% due to operational safety measures were plugged in this maintenance stop. No cracking processes were observed in the tubes, but corrosion process with mass loss.

3.4 Internal Rotation Inspection System

Figure 11 shows the schematic drawing used to perform the ultrasonic thickness measurement test using the IRIS (Internal Rotation Inspection System) technique on the tubes of the heat exchanger. The representation is at a percentage level of loss of wall thickness of the tube, where a loss of 100% indicates that the tube has lost containment and is perforated and a percentage of 0% indicates that the tube is without thickness loss.

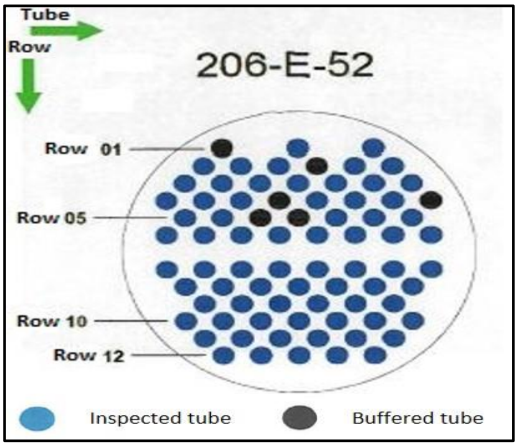

Figure 11: Schematic drawing of the ultrasonic test for measuring thickness in the tube.

The concentration of tubes with thickness loss was the highest in the region of the upper pass of the tube bundle, a region that is located at the entrance of H_2S + DEA fluid, on the outside of the tubes; where the test indicated the origin of the corrosive process, that is, corrosion from "outside to inside", due to the aggressive action of the fluid in the metallic material of the tubes. The greatest corrosion damage in the region of the upper pass of the tube bundle was aggravated by the impingement caused by the entrance of the fluid. IOFA *et al.* [19] demonstrated that H_2S at low pH or HS^- at medium pH forms a monolayer film through chemi-sorption on iron. The sulfide film formation involves the formation of cubic iron sulfide (FeS), tetragonal mackinawite (FeS_{1-x}), and hexagonal troilite [20]. The iron corrosion mechanism is described by reactions:

$$Fe\,(s) + HS^-\,(aq.) \leftrightarrow FeHS^-\,(ads) \tag{2}$$

The anodic process is described by

$$FeHS^-\,(ads) \leftrightarrow FeHS^+\,(ads) + 2e^- \tag{3}$$

The positively charged iron hydrosulfide ion is hydrolyzed.

$$FeHS^+\,(ads) + H_2O \leftrightarrow Fe^{2+}\,(aq.) + H_2S + H_2O\,(aq.) \tag{4}$$

The adsorbed $FeHS^+$ (ads) can dissolve or form the mackinawite film.

$$FeHS^+\,(ads) + H_2O \leftrightarrow Fe^{2+}\,(aq.) + H_2S + OH^-\,(aq.) \tag{5}$$

$$FeHS^+\,(ads) + xFe^{2+}\,(aq.) \leftrightarrow Fe_{(1+x)}\,S_s + H^+\,(aq.) \tag{6}$$

At pH values lower than 6.5, which is the case with the DEA and H_2S solution that has pH 5, mackinawite has a high solubility and reaction (5) dominates, resulting in enhancement of the corrosion rate [20]. The deleterious action of the bisulfide ion was observed, attacking the protective layer of chromium and iron oxide of AISO 316L steel, probably forming iron sulfide parallel to the iron and chromium oxides and causing the localized corrosion observed in the Figures 7 and 9.

The Internal Rotation Inspection System results for the Row 3 and Tube 1 are shown in Figure 1**2**.

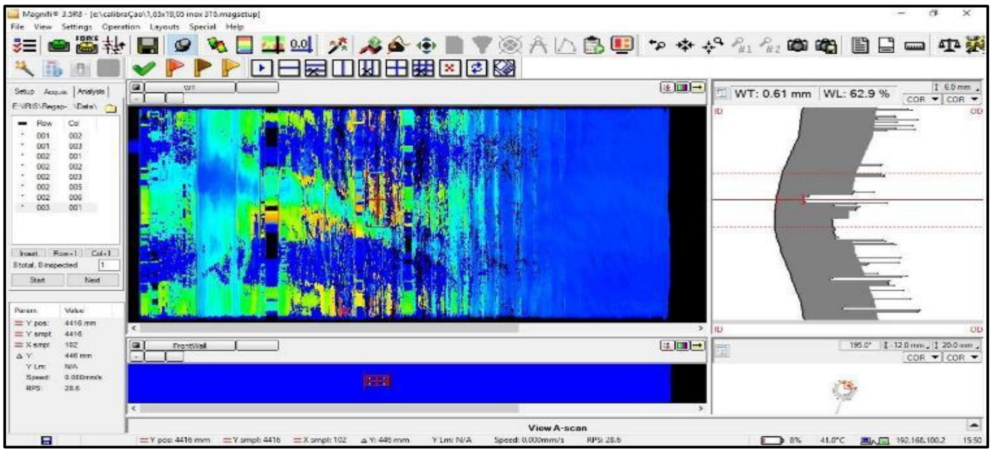

Figure 12: Internal Rotation Inspection System results for the Row 3 and Tube 1.

The test indicated the location of each tube of the bundle, concerning the total length of 6,096 mm. There was the greatest thickness loss or hole along the tube length. It also shows the remaining wall thickness found and, finally, the percentage of thickness loss, considering the wall thickness used in the equipment design, or about 1.65 mm. Regarding the Eddy current test, an increase in the corrosive process of the material was identified, with more significant thickness losses and even with the detection of tubes with loss of containment. With these results, the action implemented was the complete replacement of this tubular bundle. Again, no cracking processes were observed in the tubes, but a corrosion process with mass loss.

3.5 Metallography

After analyzing the metallographic structure of the samples extracted from the tubular beam, of specification ASTM A-213 Grade 316L and applying the recommendations of the standard ASTM A-262 practice-A, the presence of a typical structure for this steel was observed, with well-defined grains and grain boundaries, and the presence of twins as show in Figure 13. There is no evidence of intergranular corrosion. The observed corrosive process was caused by DEA and H_2S fluid that attacked the stainless steel surface of the tubular beam of the heat exchanger.

Figure 13: Optical micrograph of ASTM A213 Grade 316l.

The analysis of the chemical process involves treatment with DEA aimed at purpose of removing H_2S from the chain by placing it inside the specifications related to corrosivity and sulfur content. The process is based on the fact that ethanol-amine solutions (mono, di and tri), at a temperature close to environment, combine with H_2S, forming stable products. The products formed, when subjected to heating, are decomposed, regenerating the solutions, and releasing H_2S and or CO_2 which are sent to suitable storage tanks and sent to sulfur treatment units. DEA is subjected to a process from progressive heating to the regenerating tower, where H_2S is released where medium pressure water vapor is condensed.

The presence of DEA above the concentration of 20 wt.% in the aqueous stream external to the tubes of the heat exchanger in association with contaminating products in the stream, such as H_2S dissolved in the stream causes degradation of the passive layer of chromium and iron oxides/hydroxides, producing and removing the iron sulfide layer that forms on the surface of the metal, thereby exposing the metal to corrosive attack again [12]. In this case, the surface film is iron and chromium oxides/hydroxides and iron sulfides. In this case study, amine degradation products were not identified such as heat stable amine salts and other contaminants, but in richer amine solutions such as the solution in this case the amine degradation can occur.

In general, an inspection plan for the detection and monitoring of this type of damage mechanism involves visual examination and ultrasound thickness measurement. Ultrasound scans or profile radiography are used for external inspection, corrosion monitoring can also be achieved with corrosion coupons and/or corrosion probes, monitoring should target the hot areas of the unit such as the reboiler feed and return line, the hot lean/rich amine piping, and the stripper overhead condenser piping and fouling of exchangers and filters can be a sign of corrosion problems on the unit. The installation of the washing water system diluted the concentration of the DEA stream to levels below 5 wt.%.

4. CONCLUSIONS

The monitoring tests used confirmed a localized stainless steel corrosion with loss of containment by holes in tubes of heat exchangers of top systems of tower condensers rectifier of the hydrotreating unit of oil refineries. The internal and external visual testing of the tubes, determination of the chemical composition of the tubes, Eddy current tests, thickness measurement tests by ultrasound in the tubes and metallographic analysis were successfully used to evaluate the corrosion process in tubes of a heat exchanger from the top system of the regeneration tower of the hydrotreatment unit in an oil refinery. The fluid in the outer region of the tubes is a mixture of H_2S + DEA in an aqueous medium and in the inner region it is the cooling water, the tubes are composed of austenitic stainless steel of the ASTM A213 Grade 316L specification. The localized corrosion process occurred on the outside of the tubes, mainly in the region of the upper pass of the tube bundle, a region that is located at the entrance of H_2S + DEA fluid.

The results of the tests indicated a considerable loss of thickness of the wall of the tubes originating in the external region of the tubes, due to the corrosion mechanism by DEA and H_2S present in processes with concentration above 20 wt.% of DEA. No cracking process was observed in the tubes, only blister-type corrosion with the occurrence of holes located in the material. The tubular beam was replaced during the scheduled maintenance stop, and a routine for measuring and monitoring the DEA concentration in the flow upstream of the heat exchanger was proposed. The installation of the washing water system diluted the concentration of the DEA stream to levels below 5 wt.%.

5. ACKNOWLEDGMENT

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