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Corrosion Study of API 5L X60 Gas Pipelines Steels in NS₄ Simulated Soil

A. Benmoussat and M. Traisnel

Abstract External corrosion and cracking are the major threats and the principal mechanisms of buried pipelines deterioration. Indeed they reduce the structural integrity of the transmission system of gas pipelines. Investigations carried out on GZ1 site line, in Algeria, after about 30 years of exploitation under high pressure revealed that under certain conditions the not protected surfaces in contact with the ground developed many failures by corrosion and cracking: coating failure, reductions in thickness, pitting, etc. In this study the methodology was defined initially, by an expertise of the characteristic parameters of working GZ1 line which broke due to external corrosion, and then a simulation, in laboratory, of the conditions and the corrosion damage mechanisms. We were particularly interested in the corrosive electrolytic medium choice because it can simulate NS4 soil solution and the protection system by using corrosion inhibitors containing polyphosphates. Results showed that the corrosion potential is slightly moved towards the anodic values, when the pH of soil solution tends to a light acidity. This shift goes with a clear reduction of anodic and cathodic current densities and the polarization resistance R_p decreases. In the explored temperature range and simulating soil, corrosion current density increases with temperature increase. The corrosion activation energy increases with temperature according to the soil environment. Nyquist diagrams present the same general form for corrosion according to the slightly neutral pH for various immersion times in the steel/test solution interface.

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1 Introduction

The transport of oil and natural gas fossil resources from their geographical localization to their consumption centers usually located in industrialized zones takes place by the mean of buried pipelines networks. Since 40 years, an important buried tube network has been constructed and always develops through the world [1]. The search for always increasing pipeline profitability has led to the development of high-strength and high-toughness pipelines steels and to avoid any increase of tube thickness. The transport reliability depends on soil environment interaction and the choice of pipeline material and steel protection which will avoid damage. Corrosion risks are preoccupying phenomena in oil industry. External corrosion pits and cracking phenomena are the major threats and the main deterioration mechanism of buried pipelines under coating failure and cathodic protection (CP) that can reduce the structural integrity of buried pipelines transmission system. Corrosion behaviour of low carbon steel pipeline alloys in soil environment under protection failure motivates our research. The objective is to bring a better comprehension of damage mechanisms and to reduce steel failures in service.

Carbon steels of weakly C-Mn allied type as API 5L X60 of GZ1 pipeline in Algeria [2] are protected from the external soil aggressions by a bituminous coating whose action is coupled with a cathodic protection system (minimum potential specified -850 mV versus (Cu/CuSO₄)), which aims to maintain steel in its protection field and thus, to avoid any risk of external damage by corrosion or cracking during a possible rupture of the coating. Unfortunately, the investigations carried out on GZ1 site line [3], after about 30 years of exploitation under high pressure revealed many failures: coating failure, reductions in thickness, pitting, cracking. These chemical, biochemical or mechanical failures occurred particularly in clay soils, like montmorillonite type or saline underground waters subsoil.

Steel pipelines corrode in soil by complex electrochemical processes because of different nature of soil electrolytes. Corrosion phenomena in underground conditions are still unclear, because soil is a complex material, a porous, heterogeneous and discontinuous environment constituted by mineral or organic solid phase, water liquid phase, air and other gas phases. It is necessary to examine every particular site to explain the corrosion mechanisms models. The factors that influence corrosion in soil are numerous as soil type, moisture content and the position of the water table, soil resistivity and soluble ion content, soil pH, oxidation-reduction potential and the role of microbes in soil corrosion. Soil properties depend of soil particle size distribution, organic content, mineralogical composition and structure. Soil profiles are developed from parent materials in response to factors associated with the climate. Mineral composition is a key to understanding how a soil can influence the corrosion of buried steel. Clays are among the most common minerals on earth. Most clay has notable plasticity when wet and a marked ability to adhere to surfaces. Physically, clays of the montmorillonite group such as bentonite can radically change volume through dehydration/rehydration or ion exchange. This shrinking and swelling can exert forces on structures buried in montmorillonite rich soils

leading to potentially detrimental consequences. Coarse silica sands tend to be relatively permeable, well drained, and inert. Dissolved carbonate will buffer the solution in the neutral to alkaline pH range [4]. Exposure of this saturated solution to steel surfaces rendered alkaline by electrochemical reactions induced by an effective cathodic protection system will precipitate hard white carbonate scales on the metal surface which can involve corrosion problems. The physicochemical parameters of soil including resistivity (ρ), redox potential (E), pH, salt and moisture content can be determined quickly to estimate soil corrosivity. Weight loss measurements is the most important parameter for determining soil corrosivity, but it takes a long time to obtain weight loss data. The electrochemical parameters such as corrosion current density (I_{corr}) and polarization resistance (R_p) can serve as parameters for evaluation of soil corrosivity with accuracy and ease. Electrochemical impedance spectroscopy (EIS) and potentiodynamic polarisation [5] were carried out to study the mechanism of pipeline steel soil corrosion. The likely rate of corrosion of underground corrosion can be assessed in terms of the soil pH, resistivity, temperature, and redox potential. However, the relationship between soil corrosivity and physicochemical parameters is very complex; therefore, the soil corrosivity appraised by these parameters is often unreliable. A number of probes have been developed for field measurements of these parameters. Li and Cao [6] reported the development of a new soil corrosivity probe that could be used to measure not only corrosion current density and corrosion potential of metals in soil, but also parameters of soil.

2 Materials and Methods

In this study the methodology was defined, initially, by an expertise of the characteristic parameters of working GZ1 line which broke due to external corrosion, and then a simulation, in laboratory, of the conditions and the corrosion damage mechanisms. We were particularly interested in the corrosive electrolytic medium choice because it can simulate NS4 soil solution and the protection system by using corrosion inhibitors containing polyphosphates.

2.1 Pipeline Steel

In zones where GZ1 steel pipeline are damaged (from pK = 145 to pK = 226), (pK = kilometer point) samples of corroded X60 steel and not corroded ones have been studied by micro analytical techniques. These tubes from 40" diameter are rolled and welded in spiral are posed in 1976 which the failures corrosion pits and cracks were detected. Micrographic analysis has been done by scanning electronic microscopy (SEM). Elemental composition of the samples was determined by spectrophotometric analysis type "SPECTRO RP 212" Measured values were compared to values quoted in the material test certificates [7]. Electrochemical

Table 1 Elemental composition of API 5L X-60 steel pipeline

C%	Si%	Mn%	P%	S%	Cr%	Mo%
0.180	0.0364	1.4000	0.0175	0.0131	0.0284	0
Ni%	Al%	Cu%	Ti%	V%	Sn%	Fe%
0.0182	0.0493	0.0326	0.0058	0.0061	0.0031	≈97.880

measurements, potentiodynamic polarization and impedance spectroscopy (EIS) were carried out by means of potentiostat equipment. Soil simulating solution was chosen according to results obtained of the most aggressive GZ1 environment composition.

Steel pipeline of weakly C-Mn allied type as X60 for use in gas transmission is manufactured according to the specified chemical composition and mechanical parameters. It is manufactured from a variety of materials including carbon steel and corrosion resistant alloys (Table 1). Steel, although susceptible to corrosion, is widely used because of its low cost, high strength and the ease of field makeup by welding. The use relies on appropriate design allowances and corrosion controls. It is manufactured to the API-5L specification cover grade X60 and other grades [7]. It has a specific set of mechanical parameters including: yield strength, tensile strength and toughness that pipe must comply with. Chemical composition is specified as maximum limits of four elements, i.e. carbon, manganese, phosphorus and sulphur. Composition and microstructure can vary significantly between pipes. These variations result in substantial differences in corrosion performances of pipeline steel in a corrosion regime. GZ1 tube diameter of 40 in. and a fine pearlitic-ferritic microstructure have been manufactured in Algerian Annaba steel factory controlled lamination and accelerated cooling. The refinement of ferritic grain size has been obtained by different mechanisms of hardening and precipitation based on the dislocation movement that increases elasticity limit and steel tenacity. Hall-Petch laws [8] have since been verified experimentally and explain the hardening induced by a reduction in the ferritic grain size.

$$\sigma_y = \sigma_0 + \frac{K_y}{\sqrt{d}} \quad (1)$$

σ_y – Elasticity limit

σ_0 – sum of hardening stresses based on the dislocation movement

K_y – constant expressing the grain size effect

Samples cover grades X60 within the API-5 L specifications, were obtained from pipe Algerian gas producer's society SONATRACH. Materials have been chosen as representative of tubes posed on the line in 1974 where corrosion pits and corrosion cracks were detected. Samples were cut by flame-cut from the pipe walls and the test coupons were then cut from these sections by wet sawing. The cutting process was chosen as it does not alter the microstructure and corrosion test at the coupon surface, due to its low heat input and the absence of mechanical damage. The sections were polished by emery paper of 600–2,000 grit before being tested.

2.2 Soil Simulating Solution

The corrosion is related to soil conditions in which the structures are buried. The techniques available to determine the aggressiveness of the site may include laboratory tests based on a soil chemical analysis in a specific location. Several soil samplings have been taken from various Algerian sites from which we have chosen the most aggressive composition. Soil extract was prepared according to AFNOR French norm A-05.250 P. 278.

A mass of ground is taken, then mixed with distilled water and analyzed by spectrophotometer microanalysis. The chemical composition of soil is given in Table 2. The criterion of steel aggressiveness is principally the chloride, sulphate and bicarbonate content. However, the analysis should also include the concentration of sodium, potassium and calcium ions so that an ionic balance can be struck. The test solution is obtained by reconstitution of the chemical composition of soil in a solution called "soil simulating solution". Si is the considered site. The analogue soil excavations determined some synthetic solutions named NS1 to NS4 [9]. The chemical composition is described in Table 3.

The soil chemical composition shows that the principal electrolytes contained in pipe soil environment are variable proportions of carbonates and bicarbonates according to line sites and quantities of chlorides and sulphates. NS₄ synthetic electrolytic solution was selected as soil simulating solution and aggressive solution for electrochemical measurements. The pH of NS₄ synthetic electrolytic solution is ranged between 8 and 8.5. pH measurements raised on the line showed values ranging between 6.5 and 8.5. This low value for tubes submitted to a cathodic protection and generating alkalinity by hydroxyl ions is explained by CO₂ dissolved in the electrolyte. In laboratory, a bubbling of CO₂ gas in an aqueous solution would permit to adjust the pH of the medium to pH ≈ 6.7.

Table 2 Chemical composition of corrosive soils solution

Sites	Mass (mg/l)					
	Ca ²⁺	Mg ²⁺	K ⁺	Cl ⁻	SO ₄ ²⁻	HCO ₃ ⁻
S1	94.60	56	7.6	76.9	736	117
S2	18.96	16.44	11.7	47.33	458.4	183
S3	–	–	6	7.8	74	218

Table 3 Composition of NS_i synthetic electrolytic solution of soil [9]

Composition (mg/L)	Denomination			
	NS ₁	NS ₂	NS ₃	NS ₄
KCl	149	142	37	122
NaHCO ₃	504	1,031	559	483
CaCl ₂ ·2H ₂ O	159	73	8	181
MgSO ₄ ·7H ₂ O	106	254	89	131

2.3 Electrochemical Measurements

Pre-treatment of steel samples surfaces was carried out by grinding with emery paper of 600–1,200 grit, rinsing with bidistilled water, and ultrasonic degreasing in ethanol and drying at room temperature before us. All tests have been performed at $30 \pm 1^\circ\text{C}$. Electrochemical measurements were carried out by means of Tacussel – Radiometer PGZ 301 equipment. Polarization equipments were carried out in a conventional three electrodes glass cell with a platinum counter electrode and a saturated calomel electrode (SCE) as reference with luggin capillary bridge. All tests have been performed in de-aerated solutions under stirred conditions at room temperature. Electrochemical cell used and polarization measurements was the same as described in paper [10]. The potentiodynamic polarization curves were recorded by a constant sweep rate of 0.5 mV s^{-1} . Before recording the polarization curves, the open – circuit potential was stable within 30 min. The cathodic branch was always determined first, the open–circuit potential was then re–established and the anodic branch determined.

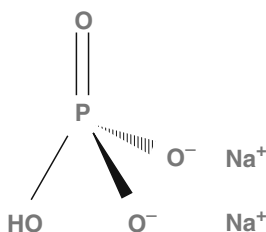
Electrochemical impedance spectroscopy (EIS) measurements were performed using Tacussel – Radiometer PGZ 301 frequency response analyser in a frequency range of 10^5 to 10^{-2} Hz with 10 points per decade. Impedance equipment is controlled by Tacussel corrosion analysis software model voltamaster 4. Square sheet steel of size ($5 \times 5 \times 0.06 \text{ cm}^3$), which exposed a 7.88 cm^2 surface to the test solution, were used as the working electrode.

2.4 Soil Resistivity

Soil resistivity (ρ) was determined according humidity values on line environment. The moisture content was determined by weight loss measurements. One weighs an earth sample is dried in a drying oven 105°C during 24 h. The weight difference between earth sample before and after evaporation is regarded the moisture content determined as the evaporated water mass. This method is not precise and does not give a constant weight after earth sample evaporation.

2.5 Corrosion Inhibitor Used

The corrosion inhibitor used is disodic Hydrogéo-monophosphate is an odourless hygroscopic white powder of chemical formula: Na_2HPO_4



The inhibitors solutions containing polyphosphates were obtained by dissolution in distilled to obtain different concentrations (10^{-3} , 10^{-2} , 10^{-1} , 5.10^{-1} M).

3 Results and Discussions

3.1 Potentiodynamic Polarization

The anodic and cathodic polarization curves are recorded on low carbon steel in de-aerated soil simulating solution at various pH and temperatures that simulates conditions of pipe soil environment.

3.1.1 pH Influence

Polarization curves in the pH range 6–8 are shown in Fig. 1. The polarization parameters values of (I_{corr}), corrosion potential (E_{corr}), polarization resistance and cathodic and anodic Tafel slopes (b_c), (b_a) are given in Table 4. For corrosion potential value ($E_{\text{corr}} = -452.8$ mV/SCE), the corrosion of low carbon pipeline steel in acidic soil simulating solution is obtained.

Results of polarization curves show that the corrosion of iron as a function of pH increases considerably at acidic environments. If values of pH decrease toward the neutral or acidic pH in the range 6–8, steel corrosion increases and polarization

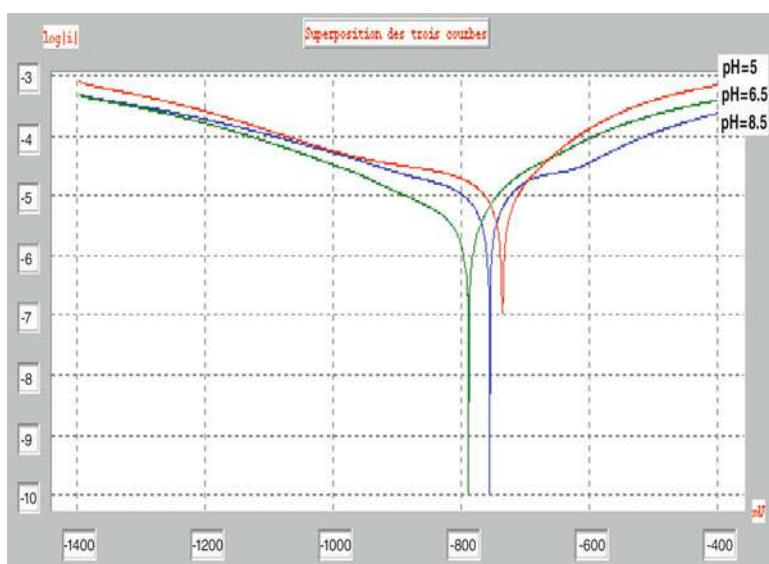


Fig. 1 Potentiodynamic polarization curves of X60 carbon steel in NS4 simulating solution of corrosive soil environment at 30°C

Table 4 Polarization parameters for the corrosion of carbon steel in soil simulating solution according to the pH variation in the range 6–8

pH (mV)	$E_{\text{corr/SCE}}$ ($\mu\text{A.cm}^{-2}$)	I_{corr} (mV.dec^{-1})	b_c (mV.dec^{-1})	b_a (mV.dec^{-1})	R_p kohms.cm ²
6.7	-452.8	3.137	177.0	190.4	6.87
7.5	-704.4	26.686	475.5	168.5	1.51
8.0	-759.3	5.715	231.7	87.2	3.54

resistance decreases. In alkaline pH from $\text{pH} \approx 8$, the corrosion of steel decreases and polarization resistance increases.

The pH of soil will generally fall within the range 4–10. Soils containing well humidified organic matter tend to be acidic. Mineral soils can become acidic due to leaching of basic cations (Ca^{2+} , Mg^{2+} , Na^+ , and K^+) by rainwater and as the result of dissolving of carbon dioxide into the groundwater. In the context of steel corrosion in soil, the passivity occurs at high pH values. In contrast to iron, amphoteric metals, such as aluminium, which are protected by oxide films, can be rapidly corroded in alkaline soils with high pH values as well as in acidic environments.

King [11] in a review of soil corrosiveness developed a monogram that combined the influence of resistivity and pH on the corrosion rate of steel pipe in soil but cautioned that the figure should only be used as a guide. The monogram ignores the influence of both oxidation-reduction potential and microbial activity, key parameters in underground corrosion. It may be applied to the prediction of corrosion rates in aerobic conditions.

3.1.2 Temperature Influence

Gas pipelines operation shows that the temperature vary between seasons, or the climatic changes and can modify the interactions between steel and the middle environment in soil. Temperature effect on the steel resistance corrosion was carried out using potentiodynamic measurements. Polarization curves in the temperature range 20–60°C are shown in Fig. 2. Polarization parameters values; corrosion current density (I_{corr}), corrosion potential (E_{corr}), cathodic Tafel slopes (b_c) and polarization resistance (R_p) are given in Table 5. The temperature can modify the interaction between steel electrode and the electrolytic medium. It modifies the electrochemical corrosion speed of metals. Steel corrosion is a function of pH and temperature. In neutral pH, oxygen reduction reaction and diffusion speed are favourable reaction with temperature and causes a reduction in solubility. In acidic pH, corrosion speed grows in an exponential form with the temperature because of the over tension reduction of hydrogen release.

In the studied temperature range, the corrosion current density increases with increasing temperature and the steel corrosion potential moves towards the negative values when the temperature increases in the studied solution. The anodic polarization curves present parallel Tafel straight lines indicating that the hydrogen

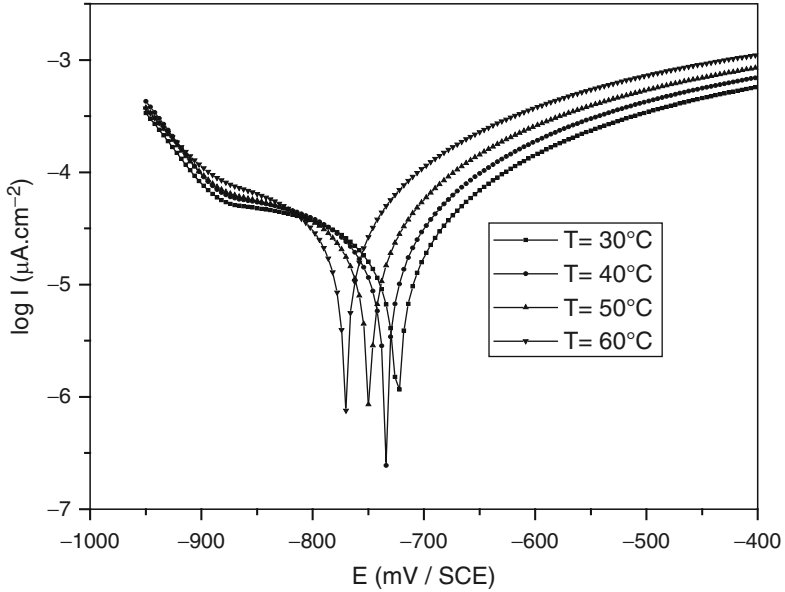


Fig. 2 Temperature influence on polarization curves of C-Mn steel in NS4 simulating solution of corrosive soil environment (pH ≈ 6.7)

Table 5 Polarization parameters for the corrosion of low carbon steel in NS₄ soil simulating solution according to the temperature (pH ≈ 6.7)

Temperature [°K]	E_{corr}/SCE [mV]	I_{corr} [$\mu A.cm^{-2}$]	b_c [mV.dec ⁻¹]	R_p [kohms.cm ²]
293	-705.0	16.86	563.4	2.59
303	-705.0	26.68	475.5	1.51
313	-715.0	33.43	668.1	1.31
323	-727.8	38.98	631.9	1.15
333	-750.0	44.17	457.0	0.913

reduction reaction to steel surface is always done according to activation mechanism in all the temperature range studied. Results of temperature effect show that the corrosion current density increases with increasing temperature in corrosive test solution. The corrosion reaction can be regarded as an Arrhenius-type process, the rate is given by the following equation:

$$\log I_{corr} = -E_a/2.303 RT + k \quad (2)$$

Where k is the Arrhenius pre-exponential constant, and E_a is the activation corrosion energy for the corrosion process. Figure 3 present the Arrhenius plots of corrosion current logarithm density vs. $1/T$. The E_a values were determined from the slopes of Arrhenius plots and are calculated to be $E_a = 13.91 \text{ kJ mol}^{-1}$. This value is low compared to the value of steels corrosion activation energy in acidic

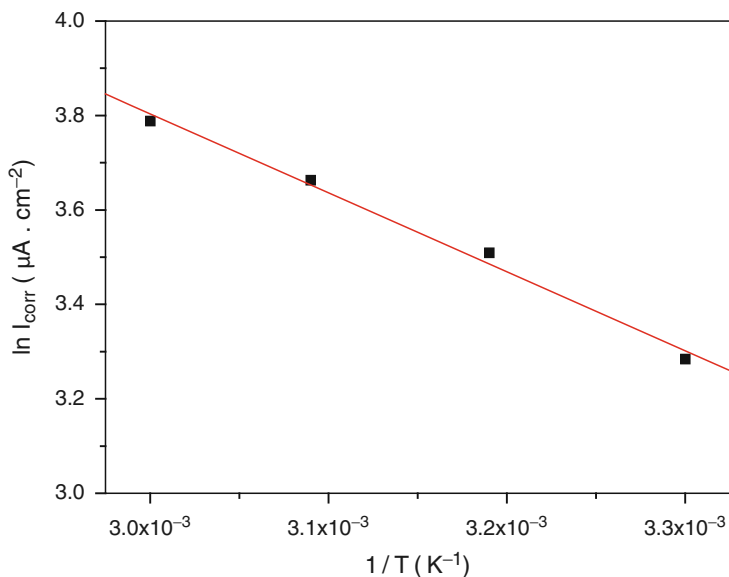


Fig. 3 Arrhenius plot calculated from corrosion current density for steel in NS4 simulating solution of corrosive soil environment (pH \approx 6.7)

environment which to reach a value of 60 kJ mol^{-1} decreases in inhibitors presence. This reduction probably was attributed to inhibitors chemisorptions on the steel surface [11]. The activation corrosion energy increases with the temperature according to the pH soil environment. The protective efficiency of external coating is function of temperature and it protective properties and decreases the activation corrosion energy.

3.2 *Electrochemical Impedance Spectroscopy*

The corrosion behaviour of carbon steel in soil simulating solution was investigated by the EIS method at $30 \pm 1^\circ\text{C}$ after immersion for 24 h. Impedance diagrams of steel/solution interface at various potentials imposed was obtained varying from the corrosion potential to the cathodic rest protection which simulates the pipes operating conditions. Figure 4 presents the evolution of Nyquist diagrams of steel pipeline obtained at the rest potential and protection potential in different pH range and immersion time of soil test solution. The impedance parameters derived from this investigation are given in Table 6. Nyquist plots displayed two impedance buckles, one capacitive buckle at the raised frequencies and diffusional loops to the low frequencies. Iron anodic dissolution and oxygen cathodic reduction phenomena are simultaneously made on surface electrode. Capacitive arc size of resistance transfer charge decreases to the slightly acidic pH. Impedance diagrams of

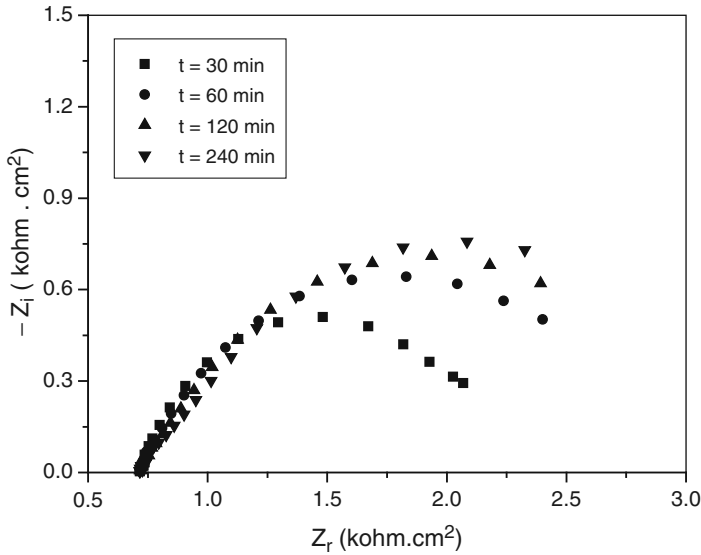


Fig. 4 Nyquist slopes for low carbon steel in NS4 simulating solution of corrosive soil environment ($\text{pH} \approx 6.7$) at different immersion times ($t = 30^\circ\text{C}$)

Table 6 Impedance parameters values of low carbon pipeline steel corrosion in soil simulating solution ($t = 30^\circ\text{C}$)

pH	R_t (ohms.cm ²)	$E_{\text{imp/SCE}}$ (mV)	C_{dl} ($\mu\text{F.cm}^{-2}$)
8.0	301.14	-700	167.90
8.0	508.40	-850	559.35
8.0	550.10	-1,000	339.62
8.0	525.60	-1,100	211.97

steel/solution interface at various potentials imposed varying from the corrosion potential to the cathodic over-protection shows that more the imposed potential is superior to (E_{corr}) values of (R_t) charge transfer resistance decreases and more the imposed potential is inferior to (E_{corr}), charge transfer resistance values decreases also but remains always superior to (R_t) value obtained with corrosion potential ($E_{\text{corr}} = -650 \text{ mV/SCE}$). Corrosion pipeline steel can be started according to operating conditions and soil environment. Immersion time influence where bare steel is in contact with the corrosive soil medium is a significant factor.

Nyquist diagrams of low carbon pipeline steel corrosion in simulating soil solution ($\text{pH} \approx 6.7$) obtained at the abandonment potential according the immersion time influence and constant temperature are shown in Fig. 5. Impedance parameters values of charge transfer resistance (R_t) and capacitance C_{dl} according the immersion time influence are given in Table 7. Results of time immersion influence shows that the charge transfer resistance (R_t) increases with increasing immersion duration in corrosive test solution. When the immersion time exceeds

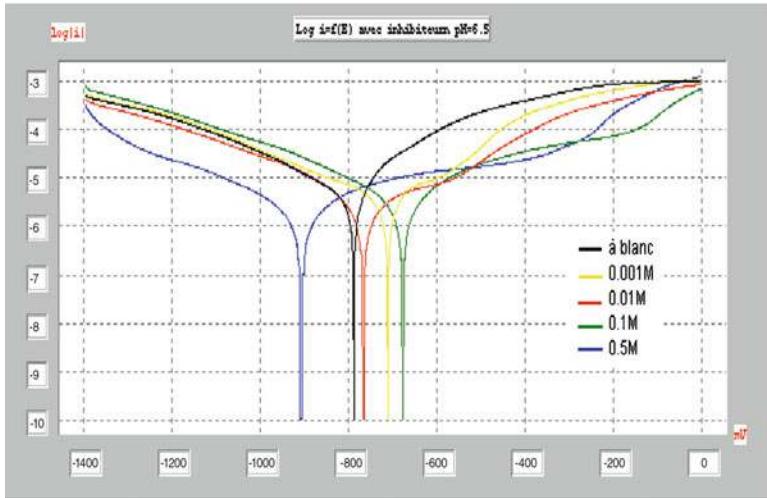


Fig. 5 Potentiodynamic polarization curves of X60 carbon steel in NS4: simulating solution of corrosive soil environment at pH = 6.5 showed the polyphosphate inhibitor influence

Table 7 Evolution of impedance parameters values (R_t) and (C_{dl}) according to immersion time of low carbon pipeline steel corrosion in soil test solution at constant temperature ($t = 30^\circ\text{C}$) and $\text{pH} \approx 6.7$

Time immersion (min)	R_t ($\Omega \cdot \text{cm}^2$)	C_{dl} ($\mu\text{F} \cdot \text{cm}^{-2}$)
30	0713.9	270.4
60	1,458.3	463.2
120	1,569.5	684.1
240	1,681.4	1,010.0

120 min steel corrosion resistance varies weakly. Capacitive arc size of resistance transfer charge increases to the time immersion increases (Fig. 4).

This result suggests the formation of protective film on steel surface whose protection increases with contact time.

4 Conclusions

Results of this investigation showed that some operating buried transmission pipeline systems will develop unforeseen pits and cracking surface corrosion problems. They are principal threats for the buried structure where the humid clay soil aggressiveness and bacterial activity appear. Steel surface must be protected by a full protective system defined by an active cathodic protection to maintain steel in potentials protection in addition to passive coatings protector aiming to avoid any corrosion interaction between steel surface and soil environment.

Future coating systems must answer the durability and reliability requirements in service in regard an economic aspect. Corrosion phenomenon is accentuated by the soils parameters influence such resistivity, pH, temperature, moisture content and chemical composition of electrolytes contained in soil. Potentiodynamic polarization curves showed that the steel corrosion increases at acidic pH environments. Corrosion current density and activation corrosion energy increases with temperature. EIS curves showed that the charge transfer resistance (R_t) increases with increasing immersion duration in corrosive test solution. Capacitive arc size of resistance transfer charge increases to the time immersion increases. This result suggests the protective film formation on steel surface. Soil resistivity decreases according to the increase in moisture content and temperature to support ionic exchange between buried steel surface and corrosive soil environment. Results showed low resistivity values in soil investigation particularly in montmorillonite clay soil characteristic of corrosive medium for buried pipeline structures. The electrochemical tests with inhibitor containing polyphosphates showed that the density of corrosion current values (I_{corr}) decreased, for a pH of NS_4 solution equal to 5, in absence of inhibitor, the density is about $7.5 \mu A/cm^2$ in the presence of inhibitor it is to $0.851 \mu A/cm^2$ with inhibiting effectiveness of more than 88%. Damage problems raise questions as the remaining safe life of the tubes. Pitting or cracking are those most to be involved in remaining life of an existing structure, because of their greater propensity to cause a reduction in the utility duration. Some of the probabilistic approaches can be applied, or are being developed, to answer the corrosion related cracking and pitting phenomena. The research will provide data for risk assessment models to be used for maintenance and operation of the pipeline system.

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