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CORROSION EFFECTS AND GREEN SCALE INHIBITORS IN THE FRACTURE MECHANICS PROPERTIES OF GAS PIPELINES

UTICAJI KOROZIJE I ZELENIH INHIBITORA NA KARAKTERISTIKE MEHANIKE LOMA KOD GASOVODA

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- green inhibitors
- fracture mechanics
- pipelines
- steel API 5L X52.

Ključne reči
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- cevovodi
- čelik API 5L X52

Abstract

The impact of the environment can cause many types of degradations such as pitting corrosion, stress corrosion cracking and sulphide stress cracking of metal structures, and one of serious problems in the oil extracting industry is the corrosion process. Recently, a number of assets failures caused by internal corrosion is recorded in the oil and gas industry. Reports have confirmed that failures are due from the effect of HCl traces. Our objectives are to use the plant extracts, such as organic corrosion inhibitors. Indeed, these natural extracts contain many families of natural organic compounds – 'green', readily available and renewable. The mechanics tests carried out on a study of anti-corrosive properties of natural products - plant origin have so far given promising results of fracture mechanics properties. The importance in this area of research is mainly related to the fact that natural products can replace present toxic organic molecules, condemned by world directives as environmentally unacceptable.

Izvod

Uticaj sredine izaziva više tipova razaranja kao što su piting korozija, naponska korozija sa prslinama, naponske prsline usled prisustva sulfida u metalnim konstrukcijama, gde je korozija ozbiljan problem u industriji nafte. Nedavno je zabeležen veći broj otkaza, izazvanih unutrašnjom korozijom, u industriji nafte i gasa. Izveštaji potvrđuju da se otkazi javljaju zbog uticaja tragova HCl. Cilj nam je primena biljnih ekstrakata, kao što su organski inhibitori korozije. Ovi prirodni ekstrakti se sastoje iz više familija prirodnih organskih jedinjenja – 'zelenih', lako dostupnih i obnovljivih. Mehanička ispitivanja, izvedena radi istraživanja antikorozionih osobina prirodnih proizvoda od biljaka, su do sada dala rezultate osobina mehanike loma koji obećavaju. Značaj u ovoj oblasti istraživanja ogleda se u činjenici da prirodni proizvodi mogu zameniti prisutne toksične organske molekule, koje svetske preporuke osuđuju kao neprihvatljive u zaštiti sredine.

INTRODUCTION

Pipelines play an important role in the industry world of gas and oil transportation. Researchers and engineers study the causes of failure and accidents to increase the safety life for as long as possible, /1/. Internal corrosion is one of the major problems encountered by the petroleum industry throughout the world and it is primarily caused by the presence of water together with acidic gas and oil, /2/. Of a total number of natural gas transmission pipeline accidents, 36% are caused by external corrosion and 63% by internal corrosion, /3/. The problem is likely to increase in severity for pipelines in oil and gas industries as the fields get older and the platforms are moved to deeper waters. Recently and a few years back, sources have confirmed a number of failures recorded in the oil and gas industry from internal corrosion, that are due from the effect of H_2S, HCl, or H_2S and CO_2 traces. The concentration of these parameters increases as the fields get older and deeper water exploration requires higher strength steels and sour-corrosion environments, /4/. Internal in-situ pipeline cleaning services will keep the integrity of oil and gas pipelines intact by means of a continuous internal protection from the aggressive environment by an inhibitor that forms a permanent barrier to internal pipeline corrosion. The pipe cleaning process can be placed in the field on-shore and off-shore. Pipeline scrapers are specifically utilized, developed, mechanically and chemically tested to prepare the internal pipeline wall along its full length prior to the unique application of a corrosive resistant. The development of the in-situ process stops any further corrosion from taking place. The system provides a continuous protective inhibitor lining at the fraction of the cost of pipeline replacement. The cleaning and protecting process by inhibitor is designed to work on steel pipeline diameters from 4 to 36 inches, and larger diameters are possible. The protecting system using the inhibitor prevents corrosion in hydrocarbon, gas, and water injection steel pipelines, and works with oil, gas, water, petroleum products, food and chemical products. It has the ability to cover pits and channel corrosion and will cover all lateral and girth welds. The beneficial effects of internal cleaning of pipelines with HCl, introduced with inhibitor, halts the damage caused by corrosion and prevents future corrosion damage. Incidents in today's petrochemical industries include hydrocarbons and their by-products that are causing internal corrosive damage to pipelines, costing billions of dollars per year. Internal corrosion leads to inevitable pipeline failure. The only way to stop internal corrosion permanently is through pipeline replacement, an internal pipeline coating system or protection using inhibitors. Inhibited acid batches are then pumped through the pipeline removing any wall corrosion and will leave the internal pipe wall surface with a near white metal finish. The inhibited acid is batched between two purpose-designed scrapers, ensuring the entire internal surface of the pipeline is corrosion free. Every batch is tested for solids and titrated to monitor the cleaning effectiveness. Once the inhibited acid shows no

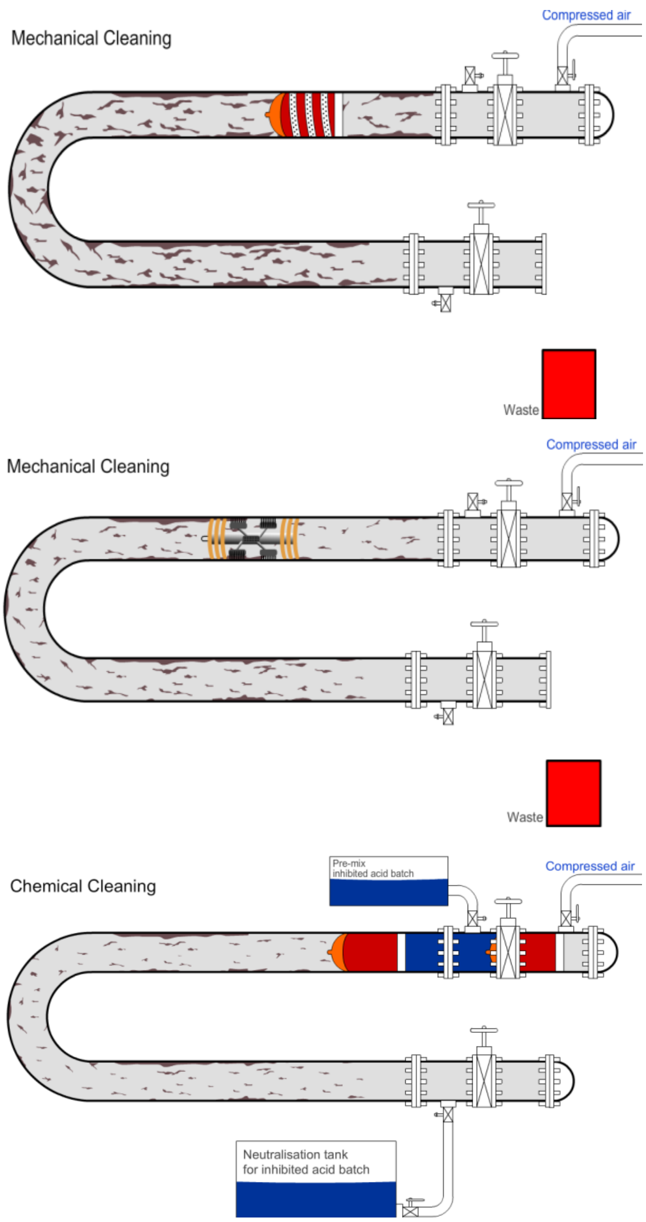

Figure 1. Internal cleaning system of pipelines, /5/.

depletion in strength, the pipeline is considered clean. The pipeline is then visually examined at predetermined inspection points for verification and total cleanliness.

Generally, there are other methods for the prevention and protection of gas and oil pipelines from internal corrosion, e.g. (i) prevent or mitigate internal corrosion by dehydration, /6/; (ii) system cleaning of corroding pipe and injecting inhibitors inside pipelines, /7/; and (iii) system injection of synthetic inhibitor inside new pipelines while in service, /8/. When the pipeline is in service, it shall be necessary to pig the line to maintain line efficiency and aid in the control of corrosion. It is necessary to remove the liquids in wet gas systems, also remove accumulated water in product pipelines, and paraffin removal and control in crude oil pipelines. When inhibitors are used in a gas pipelines, the solvents in the inhibitors evaporate, forming gunk on pipe walls that can be removed with cleaning pigs. Cleaning pigs are also used in conjunction with chemical treatment of the lines to disturb the corrosion sites and remove water, microbes, corrosion products, and food for microbes, /9/.

Figure 2. A corroded pipe (top); the same pipe cleaned and prepared by the Tomahawk™ System (bottom). The pipe is clean, dry and ready for liner application, /10/.

The protective measures taken at present to combat internal corrosion are not adequate. The methods using the synthetic inhibitor are very expensive, toxics for humans and not friendly to the environment. The use of the 'green' inhibitor is not a new idea. The initiative was made in some research, for more details see /11-13/. The new tendency is to use the 'green' inhibitor, as a solution for anti-corrosion to protect pipe steels from degradation in the oil- and oil transportation by the extraction solution from natural plants. The developed 'green' are used to inhibit corrosion in steel by the natural extracts containing many families of natural organic compounds (flavonoids, alkaloids, tannins, etc.), readily available and renewable. This method is based on using an active corrosion inhibitor as a coat on the internal surface of the steel wall, such that corrosion inhibitors will take place between the gas, oil and internal pipeline as catalyst, instead of on the corroding steel surface. Research entering the 21st century, along with people's increasing awareness of protecting the environment, a large number of research on plant leaves extracts as effective corrosion inhibitors of iron and steel in acidic media have been reported, such as henna /14/, Nypa fruticans, Wurmb /15/, Azadirachta indica /16/, Acalypha indica /17/, Zenthoxylum alatum /18/, Damsissa /19/, Phyllanthus amarus /20/, Murraya koenigii /21/, Justicia gendarussa /22/, Oxandra asbeckii /23/, Stevia rebaudiana /24/, ginkgo /25/, Reed /26/, /27/, etc… Through these studies, it is agreed that the inhibition performance of plant extract is normally ascribed to the presence in their composition of complex organic species such as (flavonoids, alkaloids, tannins, ...). This 'green' is readily available and renewable. Natural extracts contain many families of natural organic compounds, /28/. A history of the literature survey on internal corrosion and its prevention in oil and gas equipment is presented. The viability of this injection method is studied in two mechanical tests: three-point bending in static loading, and specimens with Charpy V-notch impact test. Mechanical studies carried out to evaluate the effectiveness of this injection method of corrosion inhibitors inside in-service pipelines, are helped by a literature survey on stress corrosion testing methods and an interpretation of results. Slower strain/load rate tests and sustained load tests are selected to study the changes of various mechanical parameters on different types of specimens when protected with and without corrosion inhibitors.

MATERIAL AND EXPERIMENTAL STUDY

Chemical analysis and metallography

Mechanical tests are carried out with API 5L X52 steel specimens taken from a gas transport pipe, manufactured for SonatrachTM in the transverse direction. This pipe is of outer diameter 610 mm and thickness of 11 mm. It comes from a pipeline section being 20 years in service. The steel grade of the pipe is API 5L X52. The chemical characteristics are represented in Table 1 and the mechanical properties in Table 2. Each specimen is V- notched at the centre by a manual sewing machine. However, the V-notch is most commonly used to test steels of ferritic structure. In this study, only the V-notch specimens are formed by a notch machine. The solutions are generally acidic aggressive media for metals and alloys. To overcome this undesirable phenomenon, the organic inhibitors whose mode of action is generally the result of their adsorption on the surface of the metal, are the most commonly used. Their selection depends on the type of acid, its concentration, the temperature and the metal material exposed to the action of the acid solution. The microstructure of steel, shown in Fig. 3, depicts pearlite colonies distributed in a ferritic matrix, and this is in agreement with similar microstructures obtained by others, /29-30/.

Table 1. Chemical composition of API 5L X-52 steel (wt.%).

C	Mn	P	Si	Cr	Ni	Mo	S	Cu	Ti	Nb	Al	Fe
0.22	1.22	-	0.24	0.16	0.14	0.06	0.036	0.19	0.04	<0.05	0.032	98.3

Table 2. Mechanical properties of API X52 in air, /29/.

Steel grade	σ_u [MPa]	σ_y [MPa]	Elongation, %
X52	528	410	30.2

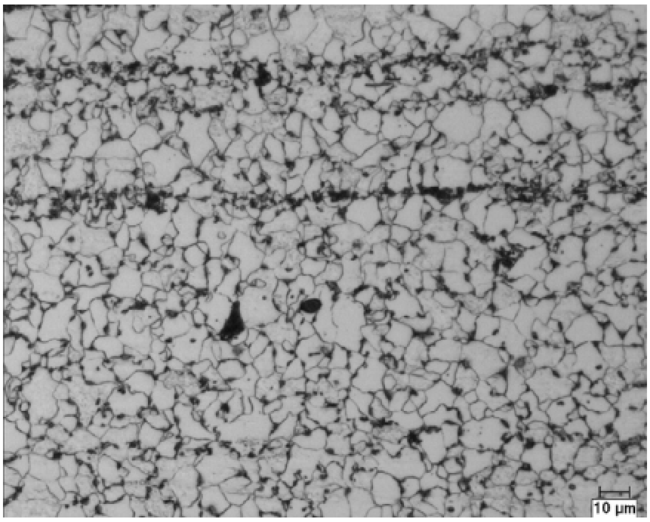

Figure 3. Microstructure of pipe section showing typical microstructure of API X52 steel grade, containing pearlite bands in a ferritic matrix, 400×, /30/.

Original and extraction of inhibitor

The aerial parts of plants in Algeria are harvested in May 2015 in the forest Sidi Maafa, in Chlef western Algeria. A voucher specimen is deposited in the Industrial Chemistry Department of the Faculty of Technology, University of Chlef, Algeria. The dried plant material is stored in the laboratory at room temperature (25°C) and in the shade before extraction, /31/.

The corrosion inhibitors are presented by the Laboratory Industrial Chemistry department of Faculty of Technology, University of Chlef, Algeria, so as to maintain constant composition throughout the experiment. Dried aloe plant leaves (10 g) are soaked in deionized water (500 ml) and refluxed for 5 h. The aqueous solution is filtered and concentrated to 100 ml. This concentrated solution is used to prepare solutions of different concentrations by dilution method. To obtain the mass of plant extract, it is dried at 100°C under vacuum in the vaporizer. From the weight of the vacuum dried liquid, plant extract is found to contain 50 mg·ml^{-1} of plant compounds, /32/.

Preparation of solutions

The aggressive solutions of 1 M HCl are prepared by dilution of analytical grade 37% HCl with distilled water. The inhibitor is dissolved in the acid solution at required concentrations in (g/L), and the solution in the absence of inhibitor is taken as blank for purposes of comparison. The test solutions are freshly prepared before each experiment by adding extract of Thymus Algeriens directly to the corrosive solution. Concentrations of the Thymus Algeriens extract are 0.125, 0.25, 0.5 and 1 g/L. We conducted tests in four-time interval of 6 hours, 5 days, 10 days, 15 days with and without the corrosion inhibitors in a volume of 100 ml, with a concentration of 1 M HCl acid and 5%, 20%, 30% of the Ruta Chalepensis, Fig. 4. We repeated this study with different concentrations of inhibitors immersed in the corrosive solution of volume 100 ml, with concentration 1 M HCl acid and 5%, 10%, 20%, and 30% of the inhibitor. The different concentrations are for obtaining the optimal concentration for corrosion inhibitors. In an interesting study, El-Etre investigated the stem extract of Opuntia (Paddle cactus) in Algeria, India, Mexico, North Africa and some parts of Europe) for the corrosion inhibition of steel in HCl acid solution, in which the extract is obtained by squeezing the stem instead of extracting with some solvent, and the juice is directly applied as the inhibitor, /33/. The aging effect of the inhibitor is also studied, which is discussed in the later section. Natural oils are one of the green inhibitors from plant sources. Pennyroyal oil is extracted from Mentha pulegium (pennyroyal mint) and studied for corrosion inhibition of steel in HCl corrosive medium, /34/. The major constituent of pennyroyal oil is (R)-(+)-Pulegone, /35/. The corrosion inhibitors are presented by the Chemical Laboratory, University Hassiba Benbouali of Chlef, Algeria, so as to maintain constant composition throughout the experiment. Dried aloe plant leaves (10 g) are soaked in deionized water (500 ml) and refluxed for 5 h. The aqueous solution is filtered and concentrated to 100 ml. This concentrated solution is used to prepare solutions of different concentrations by dilution method. To obtain the mass of plant extract, it is dried at 100°C under vacuum in the vaporizer. From the weight of the vacuum dried liquid, the plant extract is found to contain 50 $mg{\cdot}ml^{-1}$ of plant compounds, /36/. The Algerian extract leads to decrease of in the corrosion rate. The results show that the inhibition efficiency is independent of temperature, showing that the Algerian extract is an efficient inhibitor in the range of temperatures studied, /37/. We are studying the influence of acid HCl on the mechanical properties of the material in the corrosive environment, when it is immersed in the solution of volume 100 ml. We conducted tests in four time intervals: 6 hours, and 5, 10, and 15 days with and without corrosion inhibitors in the volume 100 ml, with a concentration of 1 M HCl acid and 5, 20, and 30% of the Ruta Chalepensis. We repeated this study with different concentrations of inhibitors immersed in the corrosive solution of volume 100 ml, with concentrations of 1 M HCl acid and 5, 20, and 30% of corrosion inhibitor.

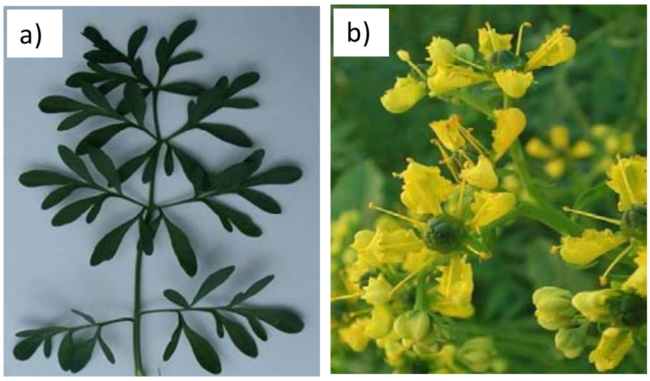

Figure 4. Photos of the plant Ruta Chalepensis: a) leaves, b) flowers, /31/.

RESULTS AND INTERPRETATIONS

Experimental study

– Study in the absence of inhibitors by dynamic test

In this section we study the influence of hydrochloric acid concentration on metal corrosion for following concentrations 0.25 M, 0.5 M, 0.75 M, and 1 M with the same immersing time. The tests are performed after 0.25, 5, 10, and 15 days of immersion. Charpy V-notch specimens, Fig. 5, are used for dynamic tests in order to study the influence of hydrochloric acid on mechanical properties. In Fig. 6 the results are obtained by varying the fracture energy as a function of concentration of the acid media for steel API 5L X-52 at 25°C. The figure shows that the variation of the fracture energy rate increases with the concentration of acid media for all flow rates, in general. The electrochemical processes of corrosion are activated with the time immersed in the acid environment.

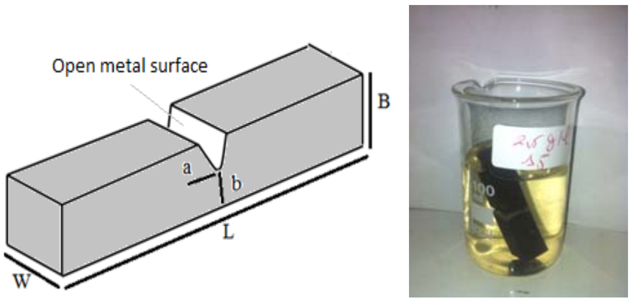

Figure 5. Specimen used in hydrochloric charging

The corrosion rate increases with degradation of the mechanical properties, because this degradation species it takes from these the species of oxidation. The acceleration stages of evolution of the energy rupture of the specimen's rate as a function of concentration of the acid media rate associated to the corrosion products /38/.

Table 3 gives the values of the fracture energies, at different concentrations of the acid, measured to rupture of specimens. Based on the results, we have shown that the fracture energy decreases by increasing the concentration of HCl acid. The decrease in the energy absorbed by the specimen is connected with the reduction of the toughness which facilitates crack propagation since the energy to achieve this propagation, decreases strongly with the concentration of HCl acid. The susceptibility of the material to corrosion is the number of protons in the electrolyte. A high proton concentration in the solution increases the aggressiveness of the environment. These results show that the dissolution of the API X52 steel is continuous in time in the hydrochloric acid.

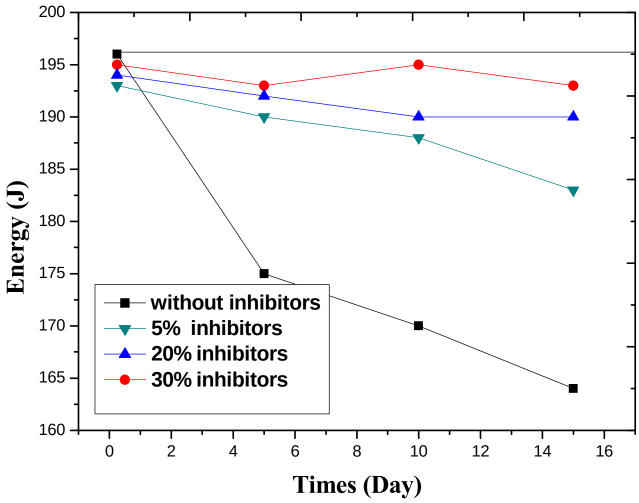

Figure 6. Fracture energy as a function of the immersion time.

Table 3. Variation of fracture energies of the steel relative to the concentration of the hydrochloric acid HCl.

Concentration (%)	0.25	0.5	0.75	1
Energy (J)	195	180	175	150

– Study of corrosion in the presence of inhibitors with the dynamic test

In this section we study the effect of the extract of Ruta Chalepensis on corrosion of API 5L X52 steel in acidic medium for different concentrations. Experimental conditions are at 25°C room temperature with a concentration of 1 M HCl acid. Measurements are made after 6 hours, 5, 10, and 15 days of immersion at room temperature. Table 3 gives the values of the energies required to fracture the test pieces measured by Charpy test for 5, 20, and 30% concentrations of inhibitor in 1 M hydrochloric acid medium. It is noted from Table 4 that the time of immersion of six hours does not involve a high change on the scale of fracture energy, even with the pure aggressive environment (hydrochloric acid 1 M). Speaking of pure aggressive environment, we did not notice a drop in the fracture energy of the specimen compared to that of the reference specimen without any degradation test. Electrochemically, it must be a corrosion phenomenon of the metal immersed in the aggressive environment that the change in mechanical properties cannot be detected at short time intervals. A decrease in the fracture energy of specimens is proportional to the time for which the specimens are immersed in the medium (with or without the inhibitor). In the inhibitor container circles, there is a stabilization of the fracture energy of specimens and this explains why the metal has kept its property (case of resilience). In the medium when the HCl acid concentration is 30%, it is observed that this stabilization of fracture energy is disturbed on the 3rd day and this is reflected in the lack of the amount of inhibitor in the aggressive medium.

Table 4. Variation of specimen fracture energy vs. immersion time with different concentrations of green corrosion inhibitors.

Inhibitor concentration	Immersion time [days]	Total energy absorbed [J]	Toughness J_{Ic} [kJ/m^2]
R_{ef}/HCl, 1 M	0.25	196	696.01
	5	175	674.84
	10	170	640.19
	15	164	638.20
Ruta Chalepensis 5% HCl	6 hours	195	730.14
	5	193	721.20
	10	195	730.14
	15	193	721.20
Ruta Chalepensis 20% HCl	0.25	194	695.23
	5	192	676.12
	10	190	680.54
	15	190	668.31
Ruta Chalepensis 30% HCl	0.25	193	702.26
	5	190	685.04
	10	188	677.81
	15	183	669.24

In the literature, it is explained that the protective film is nonhomogeneous, or the level of the metal/aggressive environment at this concentration of the inhibitor, the metal is not completely protected from the aggressive medium. Instead, the media with HCl acid concentration of 5 and 20% have a fracture energy almost equal to that of the reference specimen throughout the range of the test. This has resulted in the adsorption of the inhibitor on the whole metal surface, thus forming a layer of stable and permanent protection. It is noted in the tests that the range the fracture energy changes is relatively narrow (190 J), and this is due to the corrosion affecting the mechanical properties of the steel in a wide time interval. It is deduced that the change in fracture energy is explained by the dissolution of the metal as a result of the attack of the corrosive environment, it is practically expressed by a loss in thickness of specimens during the immersion time.

In this case the graph in Fig. 6 shows different results of the fracture energy curves for specimens in the acid media and for those in same acid with corrosion inhibitors. In the absence of inhibitors, the energy rupture drops from 197 to 165 J as a function of immersion time. However, with the presence of corrosion inhibitors a decrease of the fracture energy is noted, from 185 to 195 J. These results present the efficiency of corrosion inhibitors for API 5L X52 steel protection.

– Static test in 3-point single edge bending specimens

Three-point single edge bending specimens (SENB) are used to quantify the evolution of fracture energy as a function of the corrosion inhibitor with different concentrations by using V-Charpy specimens, as shown in Fig. 7. The V-Charpy specimens are prepared with the same procedures used in the dynamic test. Mode I notch fracture toughness is measured on SENB specimens. Four corrosion inhibitor concentrations (0, 5, 20 and 30%) are considered in the study. SENB specimens are loaded in bending (with a span of 70 mm) until fracture with a cross heads speed of 1 mm/min and a critical load P_c is recorded. The values of the critical stress, obtained from the critical load for each specimen are illustrated in Table 5. Two to three specimens of 5, 20 and 30% of corrosion inhibitor are tested for each notch configuration. The test procedure allowed for measuring and recording the instantaneous applied load, and mid-span deflection with time.

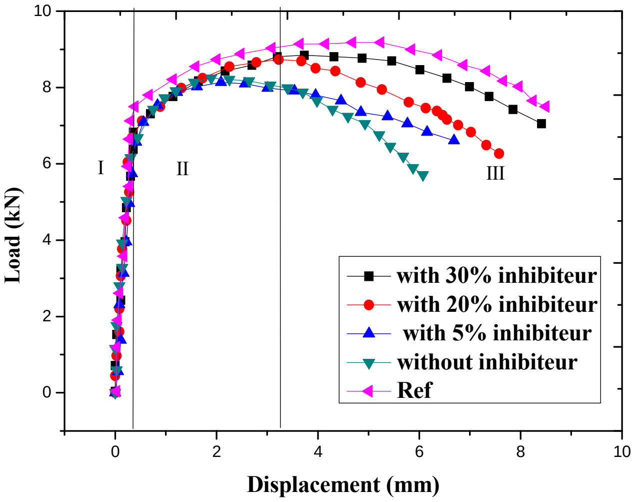

Figure 7. Fracture energies according to different concentrations in the Ruta Chalepensis model for SENB specimens.

Table 5. Load as a function of displacement for specimens immersed in different concentrations of green corrosion inhibitors.

Specimens	Max. load (kN)	Load drop, %	Displacement at max. load (mm)	Displacement loss, %
Reference	8.241	--	4.92	--
blank (HCl ,1 M)	7.21	12.51	2.25	0.54
with 5% inhibitors	7.412	10.06	2.12	0.56
with 20% inhibitors	7.812	5.20	3.39	0.31
with 30% inhibitors	8.13	1.34	3.53	0.28

Results obtained for load as a function of displacement for API-5L X-52 steel in media acid HCl with and without corrosion inhibitors are shown in Fig. 7. The corrosion rate also increases in the absence of corrosion inhibitor with time in acid media, however, in the presence of corrosion inhibitors with acid media the minimizing of corrosion is noted in the last figure for all percentage of inhibitors, 5 to 30%.

This increment in the corrosion rate is due to the presence of the ions, mainly H+ coming from the HCl, these ions accelerate the reduction and oxidation reactions between the steel and acid media. When the steel becomes very corroded it is brittle and easy to fracture. In the absence of inhibitors the maximum load at breaking is 7.21 kN, Table.5. However, in the presence of inhibitors, the observed evolution of maximum load is 7.4 kN, 7.8 kN, 8.13 kN at each concentration of inhibitors 5%, 20%, 30% as shown in Table 5, when the efficiency of the corrosion inhibitors is cured out. Bending tests are performed on 5 samples of identical geometry, where a blank sample is used as a reference, and the other four are immersed for different times in hydrochloric HCl solutions with different inhibitor concentrations. The experimental tests are used to obtain the curves of the applied load as a function of displacement. The curves give us critical maximum load values and the corresponding distance for each specimen. These figures show that the shape of the curve load/displacement depends on the concentration of the inhibitor in the aggressive environment. Thus, as already illustrated in Fig. 7, the three-point bending behaviour results in three phases, I, II, and III. In phase I, we have a linear load increase. however in phase II, this phase undergoes a non-linear load increase to reach a maximum value, while at the level of phase III, the load decreases until the specimen breaks. These curves show the degradation of maximum load as a function of concentration of inhibitors, and this results is summarized in Table 5. According to the results of this experimental study, the maximal loads in the presence of corrosion inhibitors are observed from fractured specimen tests corresponding to the 30% inhibitors, Table 5, with 1 M HCl. So this result concludes the efficiency of the 30% green inhibitors, to be more than 20% and 5% green inhibitors with 1 M HCl medium.

CONCLUSION AND PERSPECTIVES

In this study, a natural compounds extract from a plant known as Ruta Chalepensis, as a Green Inhibitor, is successively synthesized and characterized using various electrochemical and mechanical methods. Changes in mechanical properties, with dynamic and static tests, are used to study the inhibitor effect on API X52 steel and the corrosion of mild steel in 1.0 M HCl solution. This compound exhibited excellent inhibition performance as a mixed-type inhibitor with different concentrations. In general, the acidic corrosion of mild steel strongly reduces the ellongation and maximum loading. The inhibition efficiencies increase with inhibitor concentration, as used at ambient temperature in static and dynamic testing. The Green Inhibitor acts as an efficient corrosion inhibitor in 1.0 M HCl and exhibits maximum inhibition efficiency of 97%, forming a good protective film on the steel surface.

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