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Environmental stress cracking of chemically aged HDPE-100 gas pipe under impact fracture mode

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Abstract

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High-density polyethylene (HDPE) pipes are widely employed for urban gas, water and sewage transportation networks because of economic considerations and very satisfactory lifetimes. As they are straight forwardly designated for numerous hostile conditions, the stress cracking resistance needs to be investigated in combination with ageing in controlled environments. This study is set to investigate long-term sorption experiments followed by Charpy fracture tests using the same arc-shaped specimens machined from HDPE pipe. Chemically ageing media are deionized water (DW) and both hydrochloric (HA) and acetic acids (AA). For the first 4 weeks, it is found that % weight change increased drastically for HA medium. Then, for the next 30 weeks period, the inverse situation took place as the % weight change for HA diminished tending nearly to zero while it augmented for DW and AA environments. Within the first week, the impact fracture energy of HDPE decreased for all media and then increased substantially for both acids well above the as-received (AR) reference line. Charpy energy experienced a monotonic decrease for DW as swelling enhancement is promoted in the absence of plasticized material. After 7 months ageing, both acids show much higher Charpy energies than AR and DW specimens and a substantial increase in ductility. The resilience changes with respect to AR tests are respectively +110%, +75% and -25.8% for HA, AA and DW. It is accepted that strong acids (i.e., HA) easily deplete antioxidants and carbon black mixed with the resin. The combined effects of both mechanical and chemical processes can serve a more representative durability studies of HDPE pipes including ESC phenomenon.

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1. Introduction

Numerous methods have been developed to characterize the mechanical resistance of high-density polyethylene (HDPE) pipes based on diverse approaches [1-4]. Some of these methods have been suitably worked out and then adapted to evolve towards standard tests used by manufacturers in order to guarantee safety, quality and the best possible durability for the products. However, mechanical resistance is not the only assurance for HDPE pipes conveying water or extremely dangerous fluids such as natural gas in urban areas. The effect of the

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environmental service conditions must be included in order to obtain the best assessment taking into account the limitations to which plastic pipes are truly subjected to. For this reason, customary mechanical tests and their standards devoted to plastic pipes are extended to account for new design constraints, material conditioning and environmental factors during global durability characterization [5,6]. For polymers, various mechanical tests have been extended to such influence commonly known as Environmental Stress Cracking Resistance (ESCR) [7].

A number of mechanical tests are frequently used to determine the performance of polymers under impact conditions including Charpy, Izod and Falling Weight Impact. Today, Charpy fracture energy is recognized as a product property and a ranking basis for plastic pipe materials. Indeed, the superior performance of HDPE over MDPE (Medium Density Polyethylene) pipes is deduced from Charpy fracture tests, performed at 20°C and 1.6 m/s, which showed 15 and 24 kJ/m² respectively [5,8]. Also, it is established that impact loading based on ISO 17281 standard is the best example to obtain semi-brittle fracture in HDPE which is the key mechanism that resists crack propagation. Alternatively, the Charpy test described in ISO 179 is intended to measure toughness reflecting a combination of brittle and crazing mechanisms at the maximum force together with the absorbed energy by shear yielding [9,10]. The comparison between fracture mechanics-based analysis of instrumented Charpy impact test data for a set HDPE pipe grades and data of the conventional Charpy impact test concluded that the latter is established as a robust reproducible measure of the resistance to rapid crack propagation (RCP) [9]. Likewise, a formal brittle fracture mechanism is also revealed when analyzing semi-crystalline polymers pipes subjected standard impact tests and low temperatures [9,11].

ASTM-D1693 is a standard test method for Environmental Stress-Cracking (ESC) of ethylene plastics as it aims determining their vulnerability to Slow Crack Growth (SCG) in contact with chemical agents [12]. Usually, it uses notched bent strip (or U-shaped) specimens immersed in a controlled environment. However, a careful review of the specialized literature relevant to ESC of HDPE pipe does not show any experimental work using arc-shaped specimens chemically aged and tested under impact mode. PE pipe arc-shaped specimens have been mentioned in published studies dealing with standardization and mechanical aspects faraway from chemical ageing effects [13-17]. For instance, it is proposed for SCG a quantitative relationship between the fibrillar texture and corresponding applied load [13]. Likewise, few approaches are developed for radial impact plane strain fracture toughness parameters based on GIC which is characterized by the elastic critical strain energy release rate or J_{1C} which is characterized by the elasto-plastic J-integral. These toughness measurements are obtained from non-standard polyethylene arc-shaped specimens under either bending or tension modes [14-16]. Distinctly, the evolution of GIC in the radial direction is also discussed using Charpy fracture energies measured from machined arc-shaped specimens with variable HDPE pipe thicknesses [17].

The utmost risky situation causing irreversible property degradation and durability loss for HDPE pipes is ESC phenomenon as it is induced by various environmental reactions in the presence of effective external mechanical and/or thermal loads [18,19]. Depending on the nature and the possibility of amalgamation, aggressive agents may be acting alone or mutually. The most encountered ageing environments leading to HDPE degradation are frequently triggered by thermal effects, humidity or mineralized waters, hydrocarbons and fuels, alkali and acidic solutions, solvents, oxygen, chlorine, UV rays and living micro-organisms [6,7,18,19].

In the case of acids, several studies have shown the generally adverse effects on both structure and on physical properties of HDPE and polypropylene (PP) as shown in Table 1a for the case of transportation pipes [20-24] and in Table 1b for others specific applications [25-28].

Table 1a. Literature survey of HDPE pipe interactions with sulfuric and hydrochloric acids

| HDE Product | Testing Conditions | Aging time (d) | Observations | Ref. |
|----------------|--|----------------|--|-------------|
| Pipe | 33% H ₂ SO ₄ , ATR FTIR, 75°C | 30 min. | Structural changes identified by ATR FTIR, Formation of a sulfonic group and olefinic initiation, H ₂ SO ₄ attacks and destroys carbonyl impurities Probably incorporated during polymerization. | [20] ; 1985 |
| Gas Pipe | 20% H ₂ SO ₄ , Tensile, 25°C | 90 | H ₂ SO ₄ solution affects the HDPE filament structure, Decrease of E and ε _y by 58.5% and 32% respectively, Across pipe wall, lowest E values are at outer layers. | [21] ; 2009 |
| Pipe, Grade 80 | 20% H ₂ SO ₄ , Tensile, 25°C | 7 | External, intermediate and internal layers are considered, Values of E decreased by 60%, 58% and 56% respectively, Increase of ultimate strain ε _f across the pipe wall thickness. | [22] ; 2013 |
| Pipe | Mixture: H ₂ SO ₄ /SO ₄ ²⁻ Tensile, 25°C, pH=1 | 168 | Sorption experiments: HDPE shows a Fickian diffusion, Sorption rate: 0.008%; hydrolysis is invoked, Increased σ _y (+26%) and significant decrease in ε _y (~74%), Both crystallinity and density are augmented. | [23] ; 2014 |
| Pipe | HCl, (5,10, 20%) Roughness, DSC, 25°C | 129 | increase of roughness with acid concentration, Crystallinity drop (~ 33%) with acid concentration, Important loss of fracture strength. | [24] ; 2015 |

Indeed, damage degrees, following exposure to H₂SO₄ or HCl acids over variable ageing periods, are complex and multifaceted [27,29]. Significant alterations of HDPE structure together with preminent increases and/or decreases in mechanical and thermal properties are observed. When mechanical loads, thermal effects and exposition to chemically aggressive conditions are combined, resulting polymer degradations and service lifetime reduction may become even more significant [20,25-28].

HDPE pipe networks for transmission and distribution of fluids fall in the category of fluid-polymer interactions. This infers that physico-chemical phenomena such as diffusion, surfaces, and solubility are unquestionably implicated somehow and instigate certain negative effects on product performance. The solubility parameter (δ) approach as defined by Hansen is required to explain sorption observations. Such parameter typically includes the effects of 3 cohesive forces, i.e., dispersive, polar and hydrogen bonding forces [29,30]. It is understood that whenever the solubility parameters (based on dispersive forces) of both the polymer and an ageing liquid are closer, environmental stress cracking (ESC) becomes possible. Hansen’s work [30] summarizes diffusion coefficients measurements in polymers for different concentrations, anomalous type absorption rates, reporting concentration

profiles for anomalous diffusion involving surface resistance and explaining water blisters formation in polymers due to saturation. On the other hand, Hansen’s approach is also utilized to characterize physical affinities (i.e., solubility, chemical resistance, permeability...) for polymers, surface wettability and ultimately to correlate ESC occurrence in polymers using the relative energy difference number [30].

Table 1b. Literature survey of some specific cases involving HDPE or PP in interactions with sulfuric or hydrochloric acids

| HDE Product | Testing Conditions | Aging time (days) | Observations | Ref. |
|--------------------|---|-------------------|--|-------------|
| PE coating | 10% HCl, thermal expansion, 50°C | 1 | Effects of H ₂ O and 10% HCl are not significant on melting, Small increase in linear thermal expansion coefficient. | [25] ; 2019 |
| | 30% H ₂ SO ₄ , Room T° | 7 years | Liquid accumulation at polymer liner surface, Plasticization effects take place, Observed damage due to surface pulverization. | |
| Liner coating | H ₂ SO ₄ 50,70,80 and 90%, Heated up to 70°C. | | Increase in melting temperature (aging in 50%), Crystallization degree increase (hot acid), Melting point gradually decreased as high concentrations affect the molecular structure. | [26] ; 2021 |
| | H ₂ SO ₄ 70 - 90%, FTIR, heated 70°C | 20 | Detection of unsaturated double & triple bonds, Presence of carbonyl groups, Molecular structure is affected by high acid concentrations, Damage : Chain scission, dehydrogenation, oxydation. | |
| HDPE Micro-Plastic | H ₂ SO ₄ 70°C FTIR | 7 | Chemical changes: color alterations of white and black, Morphological deformation and surface sulfonation, No changes or new signals in the FTIR spectrum. | [27] ; 2022 |
| | | | 3.5% crystallinity decrease after HCl treatment, | |
| PP sheet | 6% HCl, Drying: 70°C for 3h | 60 | Overall PP polymer orientation: it becomes less orderly, Tensile tests: expression of more brittleness after exposure, 5.6% reduction in tensile strength. | [28] ; 2018 |

The objective of this study is to investigate simultaneously acidic liquid sorption and Charpy fracture energy of HDPE pipes. Specifically, the proposed testing specimens are non-standard arcs directly machined from an HDPE gas pipe [14-17]. The effects of ageing in deionized water and acidic environments on sorption and fracture energies are discussed for different periods of immersion.

2. Experimental Procedures

This section presents as-received (AR) material characteristics, sample preparation and both sorption and Charpy testing conditions. The raw material is a high-density polyethylene extruded by CHIALI Co. (Sidi Bel-Abbes, Algeria) as HDPE-100 gas pipe. It should be noted that it is a very resistant pipe and is manufactured to last in service well above the 100 years limit. Its dimensions are 200 mm in OD with an SDR of 17.6 and it is pigmented with carbon black. Table 2 summarizes its typical characteristics.

Table 2. Some physical and chemical properties of HDPE-100 gas pipe material

| Property | Limit values | ISO Standard |
|--|--------------|--------------------|
| Density, (g/cm ³) | 0.95–0.98 | 1183 |
| MFI at 190°C; 21.6 kg, (g/10min) | 0.75 | 1133 |
| Carbon Black content, (%) | 2.0 – 2.5 | 6964 |
| OIT at 200°C, (min.) | ≥ 20 | 11357-6 |
| Elastic Modulus, (MPa) | 550 – 1460 | |
| Yield Stress, (MPa) | 20 – 30 | 527-1 |
| Elongation at break, (%) | ≥ 350 | |
| Toughness, (K _{IC} , MPa.√m) | 2 – 5 | 13586 (ASTM E-399) |
| Shore D hardness, (at 20°C) | 59 | 868 |
| Resistance to cracking in surface-active environment, (mm/day) | ≤15 | (ASTM D-1693) |
| SCG Resistance, (Notched tube, 80°C, 165 h, 8.6 bars; thickness > 5mm) | No break | 13479 |

Arc-shaped specimens are prepared directly from the pipe in order to preserve most of the specific morphological variances and residual stresses imparted by the extrusion manufacturing process. Cutting is accomplished using a TOKADA 16D20 lathe at a cutting speed of 380 mm/min. while the 2 mm deep V-notch is milled on a M-HURON 55 milling machine at 486 mm/min. The arc width is 24 mm and the notch is sharpened with a one-pass razor blade (Fig. 1a, 1b) [31] Abir. Other details on machining and testing are available elsewhere [17,21,22,24].

The conditioning environments are presented in Table 3. These are (1) deionized water (DW), (2) 5% acetic acid (AA: CH₃COOH) and (3) 20% hydrochloric acid (HA: HCl) [32]. The ageing durations are 7, 14, 28 and 210 days at laboratory condition (atmospheric pressure and ~ 22°C). All specimens are weighted using an OHAUS electronic scale (max. 220 g; ±10⁻³) and then conditioned in sealed glass containers within a controlled laboratory chamber (Fig. 1c).

The tests are carried out on a ZWICK 5113 Charpy impact tester controlled with a testXpert© Software. The device complies with the standards DIN EN ISO 179, DIN 50115, DIN 53453 and ASTM D 6110-97 for Charpy tests, while for impact tensile tests, it fulfills the requirements of the standards DIN EN ISO 8256 and ISO 8256 for (Fig. 1d).

In this specific case, two methods may be considered to study HDPE ductility: (i) % cross-sectional area reduction from arc-shaped specimens and (ii) % strain at failure from standard tensile testing [33,34]. Strain failure may also be considered from HDPE filaments tensile data, available in literature [21]. Fracture surface areas measurements are achieved using the free and open-source software “ImageJ” [35]. It is mostly employed for image treatment to assess differences as they appear in the aftermath of an altering event in terms of surface area and

pixel value statistics. For the present situation, ImageJ is used as a tool for fracture surface analysis from low magnification digitized pictures.

Table 3. Number of tested specimens and characteristics of conditioning liquids

| Medium | Chemical formula | Nb spec. | Manufacturer | [Conc.] | pH solution | Aggressiveness (Lab. T°, [32]) |
|------------------------|----------------------|----------|-------------------------------|---------|-------------|--------------------------------|
| Deionized Water (DW) | H ₂ O | 25 | SARL. AA, Ain-Smara (Algeria) | - | 5.7 | HDPE resistant for T ≤ 60°C |
| Acetic Acid (AA) | CH ₃ COOH | 20 | ETS. NAIDJA, (Algeria) | 5% | 4 | |
| Hydrochloric Acid (HA) | HCl | 20 | CARLO ERBA REAGENTS (France) | 20% | 0.5 | |

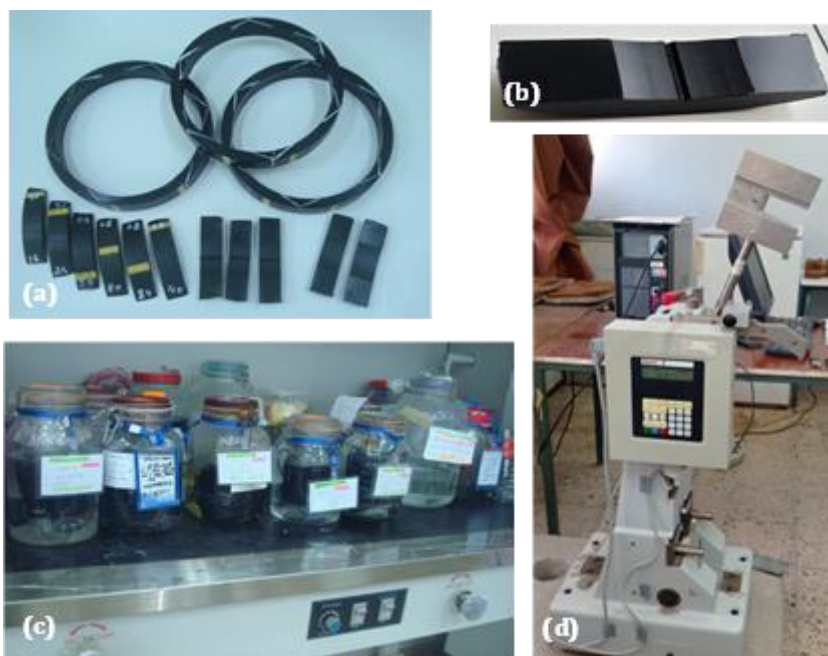


Fig. 1 (a) Cut and milled arc-specimens from pipe rings, (b) inner side notch, (c) Glass containers for aging in aggressive environments, (d) Zwick Charpy testing instrument

3. Results and Discussion

3.1. Liquid Sorption

The adopted approach in this experimental procedure is designed to have an idea about the % mass variation under the effect of liquid sorption by the HDPE arc-shaped samples before impact testing. This method allows subsequent mechanical characterization tests to be carried out on the very aged specimens. The % mass variation, presented in Figure 2 as a function of the square root of elapsed time, is calculated according to equation (1):

$$\frac{\Delta m}{m_0} = \frac{m_t - m_0}{m_0} \tag{1}$$

where m_0 and m_t are the masses measured initially and at time t , respectively.

For the environments considered in this study, different phenomena are observed during the 7 months immersion. DW medium is characterized by a relatively slow initial sorption phase (0-7 days) followed by an acceleration between 14 and 28 days and ultimately an evolution towards a form of saturation (equilibrium). This behavior is more or less similar to that of CH_3COOH , which shows an equilibrium starting the 14 days period up to 7 months. The limit sorption levels by HDPE for these 2 fluids remain below 0.04% with a concretization of a saturation-like plateau beyond the one-month period. Alternatively, the HCl case is divided into 2 parts showing a significant sorption after 14 days approaching 0.06% and followed by an accentuated desorption up to 7 months passing even below those of DW and CH_3COOH . Similar sorption data are presented in literature for DW and HCl [23].

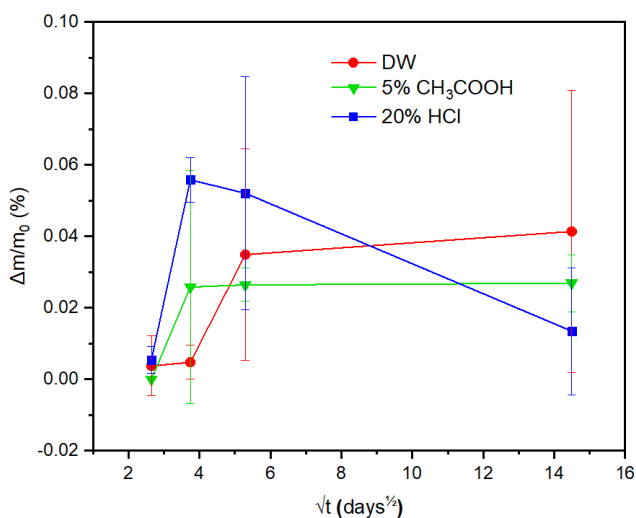


Fig. 2 Weight % change as a function of time (\sqrt{t} (Days^{1/2})) for HDPE exposed to different media: DW, 5% CH_3COOH and 20% HCl solutions

Based on Weitsman's categorization which lists different encountered sorption curves shapes, it is concluded that the 3 media considered in this study are of types (B), (LF: Linear Fickian) and (D) respectively for DW, AA and HA [36]. Generally, water sorption is identified as Type B for both polymer and composite materials. Such curve characterizes the condition of two-stage diffusion where weight gain data are related with mild fluid effects and are essentially reversible upon drying [19,36]. On the other hand, Type D curves agree with weight loss attributed to chemical and/or physical break-down of HDPE. Mainly, it is caused by leaching along interphase regions, as well as by hydrolysis, i.e., the disjoining of side groups from the backbones of the HDPE chains followed by chain scissions which is an irreversible response leading ultimately to structural failure as reported [36].

On the basis of the previously discussed solubility parameter approach, the short-term sorption order (HA, DW and AA) is explained. In the long-term, hydrochloric acid (HA) causes an increase in the crystallinity by crystallization chemistry and the resulting chain scissions together with the reorganization in crystalline clusters may play a barrier role as regards of fluid passage. In addition, the phenomenon of plasticization prevents any diffusion of HA and

a reactive polymer degradation is initiated. For other semi-crystalline polymers, energy-dispersive X-ray analysis revealed a chlorine pic after exposure to HA, indicating a chemical attack by chlorination reaction [28]. In the case of a contact between plasticized PVC and acetic acid, a simultaneous diffusion takes place as the liquid moves into, and plasticizer out of the polymer. Usually, such mass transfer is an unsteady state diffusion-controlled process [28]. Some experimental observations for HDPE interactions with acids are summarized in Tables 1a and 1b.

3.2. Impact Fracture Energy

This study examines four specific aspects associated with ESC of HDPE pipes: (i) manufacturing of non-standard arc-shaped specimens, (ii) chemically ageing in DW, acetic and hydrochloric acids for several months, (iii) measuring sorption/desorption based on actual specimens and (iv) testing under impact loading (Charpy). Such investigation is considered as a reasonable continuation for a lot of experimental work willing to introduce ESC phenomenon following the extension of the standard ISO/DIS 17281 as proposed by Niglia et al. [14] and others [15-17]. In the case of AR specimens, Fig. 3a shows the buckled final forms after impact testing. Post-failure observation shows significant deformation and an irreversible crack opening forming inconstant acute angles. The fast crack propagation covers more than 95% of specimen thickness. Total separation into two parts did not occur for AR tested specimens implying some ductility especially towards the final propagation stages (outermost pipe layers) and a substantial residual toughness left over. When using an instrumented impact tester, literature shows that for a width to crack length ratio (a/w) of 0.2, the maximum load at break attained 375 N within 1.10 ms. Moreover, increasing the (a/w) ratio, resulted in lower breaking load as the ligament is shortened [14]. However, for chemically aged ones, a few specimens are torn apart upon impact suggesting a material resistance loss and a factual evolution towards some kind of brittleness. This explanation is drawn from long-term testing of polyethylene pipes as damage mechanisms such as oxidation, polymer plasticization, chain scission and chemical degradation are found to take place lowering drastically the material strength. Measured Charpy impact fracture energies for one lot of HDPE specimens are presented in Fig. 3b.

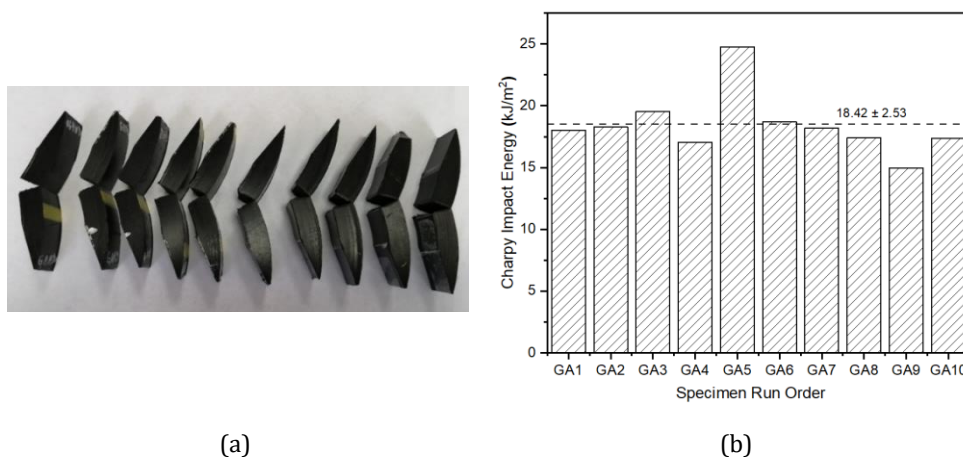


Fig. 3 (a) Ultimate shape of fractured arc-shaped specimens (GA: Gas As-received pipe), (b) Recorded impact fracture energy (Dashed line: mean value)

Good reproducibility is observed with 18.42 kJ/m² as an average value. Generally, such specific measurements based on arcs and different loading schemes are experimentally or

numerically utilized to obtain further fracture parameters such as GIC and JIC without the effects of chemical environments. Therefore, it was not possible to make appropriate comparisons with available literature [14-17]. However, when using standard specimens, Leever et al. presented closer Charpy fracture energies of 17 kJ/m² and 23 kJ/m² respectively for MDPE and HDPE at 20°C [8]. Table 4 summarizes previous impact experimental studies carried out in LR3MI laboratory on HDPE arcs provided by different manufacturers [31-33].

Table 4. Charpy fracture energy (arc-shaped HDPE specimens from local pipe manufacturers)

| Pipe Lots | HDPE Grade (Manuf. year) | OD (mm); <SDR> | Width (mm) | Nb. of specimens | Charpy Fract. Energy (kJ/m ²) | Ref. |
|-----------|---|----------------|------------|------------------|---|------------|
| 1 | HDPE-80, SARL TUNEX, (Oran), Gas (2010) | 125 | 8 | 16 | 14.53 ± 4.89 | [37]; 2012 |
| 2 | HDPE-100 TUBOGAZ Co., (Annaba), Water (2021) | 200 <17.5> | 24 | 12 | 15.95 ± 2.43 * | [38]; 2021 |
| 3 | HDPE-100K-PLAST Co., (Sétif), Gas (2020) | 200 <17.5> | 24 | 10 | 22.67 ± 1.96 * | [31]; 2022 |
| 4 | HDPE-100 CHIALI Co., (Sidi Bel-Abbès), Gas (2019) | 200 <17.5> | 24 | 10 | 18.42 ± 2.53 * | This Study |

(*) With razor cut.

Although, pipe dimensions and polymer resins are not similar, it is concluded that measured Charpy fracture energies present consistent results and allow ranking of resins.

For chemically aged specimens, Charpy fracture energy results versus elapsed time are depicted in Fig. 4. A great contrast is unveiled between the AR specimens and those aged in DW on one side and the two remaining acids (AA and CA). It is noted that the divergence in impact resistance becomes broader as the exposure time goes longer. Indeed, during the first 28 days, the fracture energy remained underneath the AR reference line for all studied media. Beyond this period, the curves diverged above and beneath the AR boundary. At 7 months of ageing, the effect of DW reduced the impact fracture energy by -25.8%, while AA and HA showed surprisingly raised values respectively by +75.3% and +110%. It should be remembered that after 28 days, a significant increase in desorption took place exclusively for HDPE specimens exposed to hydrochloric acid (Fig. 2).

For a tentative to correlate between sorption results and Charpy fracture energies as a function of ageing times, the following cases are discussed:

Case 1 (7 days ageing): On one hand, all sorption data for DW, AA and HA are below 0.005% (Fig. 2) while the Charpy energies remain rather close to each other and well below the AR ones (Fig. 4). These circumstances may imply that one week is a rather short period not fit to observe clear changes for such bulky samples.

Case 2 (14 – 28 days ageing): In this timespan, both acids (AA and HA) continue to follow the same pattern for Charpy energies while HA started undergoing a relative desorption which becomes more pronounced in the next period (Type D). After 4 weeks ageing, ranking from higher to lower, % sorption data and Charpy energies (CE) may be presented as follows:

$$\left(\frac{\Delta m}{m_0}\right)_{HCl} > \left(\frac{\Delta m}{m_0}\right)_{DW} > \left(\frac{\Delta m}{m_0}\right)_{CH_3COOH} \tag{2}$$

$$CE_{As-Received} > CE_{HCl} > CE_{CH_3COOH} > CE_{DW} \tag{3}$$

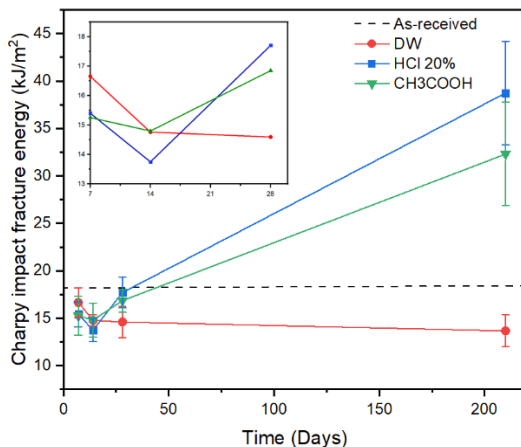


Fig. 4 Fracture Impact Energy as a function of time aged in DW, 5% CH₃COOH and 20% HCl solutions, (Dashed horizontal line refers to as-received case)

It is noticed that HDPE shows a higher sorption of HA with respect to DW and AA (Eq. (2); Fig. 2). At the same time, the Charpy energy associated with HA exceeds that of AA indicating that polymer brittleness is rapidly increasing with strong acids (Eq (3); Fig. 4). It can also be alleged that DW absorption may cause a further reduction of impact energy because of the capability of continued swelling.

Case 3 (210 days ageing): For long-term immersion, the fast desorption experienced by HA is the most striking event compared to AA and DW curves (Fig. 2). Meanwhile, DW continued to diffuse into HDPE following the stipulated Type B sorption [24,36] and causing depletion of antioxidants and other stabilizers [39]. It can be inferred that impact DW fracture energy will remain for a while below that of AR. Between 4 and 7 weeks, the AA percent weight uptake by HDPE describes a clear linear Fickian curve and remains lower than 0.027%. In addition, Charpy energies of HA and AA augmented rapidly and departed away from the AR reference line. Both observations are summarized below:

$$\left(\frac{\Delta m}{m_0}\right)_{DW} > \left(\frac{\Delta m}{m_0}\right)_{CH_3COOH} > \left(\frac{\Delta m}{m_0}\right)_{HCl} \tag{4}$$

$$CE_{HCl} > CE_{CH_3COOH} > CE_{As-Received} > CE_{DW} \tag{5}$$

Both HCl and DW environments' positions in Eqs. (4)-(5) are completely reversed, i.e., the most absorbing samples give the lowest Charpy energy and vice versa. As mentioned earlier material deterioration is occurring gradually within the HDPE structure all along with hydrolysis and readiness for ultimate failure in strong acids. The evolution towards a limit of water swelling (saturation) and its reversibility are major guarantees for the integrity preservation of the HDPE structure.

3.3 Fracture Surface Analysis

Fracture surface analysis is a necessary step in order to understand the mechanisms that led to crack initiation and its propagation during laboratory tests or under service conditions. Qualitative and even quantitative information can be extracted and may provide valuable information to formulate reasonable interpretations. Although some published work is devoted to the subject of Charpy tests on PE, there remains very little, if any, that relates to the effects of ESC caused by acidic media on HDPE-100 intended for Natural Gas transmission [8,10,13,14]. Figs. 5-7 present photomicrographs at low magnification in order to reveal different surface elementary occurrences associated with rapid crack propagation.

At this step, it is useful to remind that each picture shows the two faces of the newly created surfaces side-by-side. Scrutinizing each fracture surface and its symmetrical part should facilitate identifying macroscopic features and some of their details. Horizontal arrows indicate the crack propagation direction in both surfaces. Therefore, these figures illustrate representative fracture surfaces for AR and aged specimens in DW, AA and HA. Globally, each picture shows a notch strip, a fine razor cut trace, an initiation crack jump followed by one or more striations and a final arrest line [10,13]. The inclined dashed arrows (1) and (2) point to the limits of two main crack jumps in all pictures (Figs. 5-7). The first jump usually corresponds to a “craze zone” which is a signature representing the initiation phase of the crack extension. It is characterized by a progressively vanishing fibrillation (whitened surface elements) surrounded by a “halo” (arrow 1) [10,40].

Fig. 5 is a typical fracture surface of an as-received specimen, i.e., no prior chemical ageing is performed. The segment “ b_1 ” designates the first jump span corresponding to the initiation craze zone size at the impact moment. It is understood that tested arcs undergo a rotation at about half crack length marking another particular brittle-like crack jump (Arrow 2).

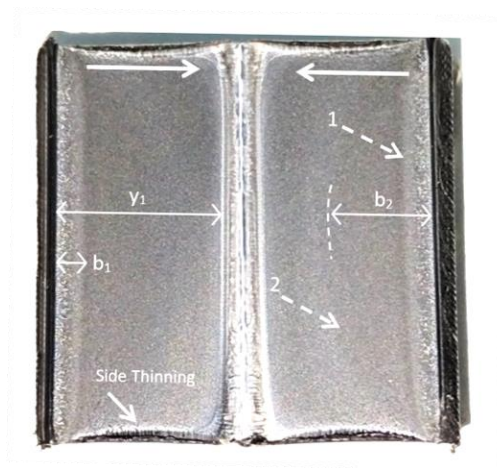


Fig. 5 A photomicrograph of global fracture surface from an unaged specimen (AR);

X~3.5; Horizontal arrows indicate CP direction

The segment “ b_2 ” designates the jump span in the brittle-like propagation zone (Fig. 5). Finally yielded HDPE material causes transverse specimen thinning, known as “shear lips”, to appear on either side of the propagating crack. The distance between initiation and final crack arrest line (large scale yielding) is denoted “ y_1 ” which will be used later for comparisons. Similarly, figure 6 illustrates the fracture surfaces of specimens aged in DW and AA. The surface marks corresponding to the effect of ageing and to crack propagation are also imprinted. The two

crack jumps are still identifiable; arrows (1) and (2) with shear lips in DW and AA. The observed fracture surface appears rough with a tendency to surface fragility probably under the effect of long-term sorption, although the quality of the external and internal surfaces of the pipe are not much affected by DW. Moreover, it should be noted that there is a weak participation of DW in the oxidation processes; that is, the structural properties of HDPE such as crystallinity and OIT are little affected [24].

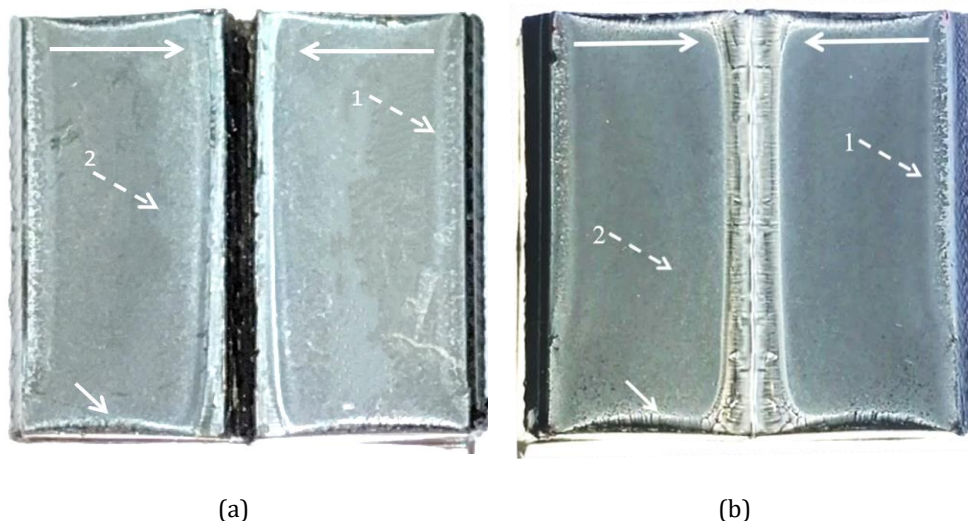


Fig. 6 Photomicrographs of global fracture surface from 7-month aged specimens in (a) DW and (b) 5% CH₃COOH (AA); X~3.5; Horizontal arrows indicate CP direction

Fig. 7 illustrates the fracture surfaces after 7 months ageing in hydrochloric acid for 2 identical specimens failed in the same conditions (HA-1 and HA-2). It is concluded that HA seems to be more damaging than AA (Fig. 6b) which showed a surface relatively similar to that of DW (Fig. 6a). The fibers within the craze zone are quite strained, the b_2 limit is rather diffuse and the side thinning is significant. The HA craze zone extent represented by distance b_1 (see Fig. 5) is larger than that of AA (Fig. 6b). On the other hand, the limits of b_2 are easily distinguishable and definitely imprinted on the surfaces (Figs. 7a-7b). The fracture surface is unquestionably of brittle nature as it exhibits localized areas with pulled out material; arrows (3); (Fig. 7a). By improving the picture contrast, the trail of the craze zone is made visible with stretched fibers and its complementary "Halo".

Also, embrittlement is much more pronounced at the crack arrest line as indicated by arrows (4). Table 5 recapitulates the quantified parameters (b_1 , b_2 and y_1) as explained in Fig. 5 and the relevant comments after 7 months of exposure. It appears that both (%) thinning and b_1 are amplified for the HA while DW and AA have similar tendencies (lower thinning and lower b_1). The b_2 parameter remains a rough approximate for both DW and AA. Nevertheless, the parameter y_1 is practically the same for all environments suggesting that the final crack arrest line occurred in comparable conditions.

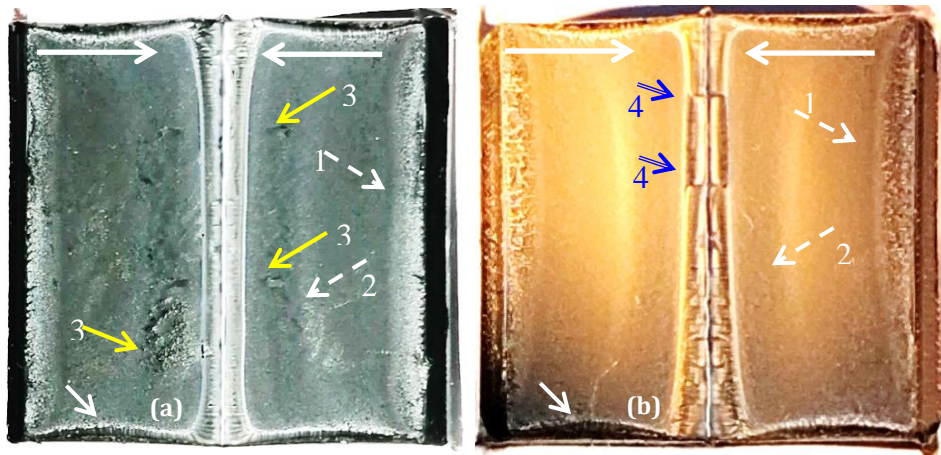


Fig. 7 Photomicrographs of two global fracture surfaces after 7 months aging in 20% HCl at different contrasts, (a) regular back and white image and (b) enhanced image (polarized light observation); X~3.5; Horizontal arrows indicate CP direction

Table 5. Thinning, bands and yielding positions from fracture surface after 7 months aging

| Aging Medium | $\Delta t/t_0$ (%) | Initiation jump, (b ₁ , mm) | Propagation band, (b ₂ , mm) | Onset yielding (y ₁ , mm) | Optical observations |
|---------------------------|--------------------|--|---|--------------------------------------|---|
| As-Received (AR) | 6.14 | 4.0 | 16.2 | 26.5 | Drawn-out fibrils within b ₁ , Smooth propagation surface, Diffuse limits of b ₂ band |
| DW | 5.83 | 3.8 | 14.4 | 26.3 | Whitened initiation line, Less dense fibrils within b ₁ , Dispersed fibrils around b ₂ , Sample breaking into 2 parts. |
| CH ₃ COOH (AA) | 5.67 | 3.7 | 20.1 | 25.9 | Highly drawn matter at initiation front, Elongated fibers within b ₁ , b ₂ : very diffuse (blurred), Substantial shear lips (yielding) |
| HCl* (HA-1) | 7.40 | 5.5 | 16.5 | 26.3 | Larger b ₁ , Highly yielded fibrils in b ₁ , Distinguishable b ₂ limits, Large surface embrittlement, b ₂ : Zone of material uprooting. |
| HCl* (HA-2) | 7.56 | 5.3 | 16.3 | 26.5 | Larger b ₁ , highly yielded fibers, Unambiguous b ₂ limits, Coarse fracture surface in the propagation zone (nearby b ₂). |

* Identical samples tested in the same conditions. HA-1 (Fig. 7a) and HA-2 (Fig. 7b).

3.4 Ductility Analysis

Since this study presents Charpy fracture data using chemically aged non-standard arch-shaped specimens, it is noteworthy to investigate the ductility associated with plastic pipe testing. It is fundamentally accepted that ductility is by far one of the most significant intensive mechanical properties for structural and pressure vessel materials. It is defined as tensile plasticity because it is governed by strain hardening rate in the last tensile test stages. As a consequence, one way to improve material ductility needs to increase strain hardening rate through microstructure modification [33,34, 41,42]. During laboratory testing and accreditation steps, ductility of HDPE pipes is a major factor for detecting early deformations due to overpressure or high mechanical loads. Therefore, this property contributes to establish safe installation and functioning conditions and also to prevent unexpected catastrophic failures.

At least, two methods are employed to approach ductility from destructive mechanical testing of HDPE pipes. Conventionally, the limiting estimate of ductility is taken as the recorded strain at failure (ϵ_f) from a standard tensile test. Alternatively, overall ductility is usually expressed whensoever achievable by percent cross-sectional area reduction (A_r) at break (eq. 6):

$$\% A_r = \frac{A_0 - A_f}{A_0} \tag{6}$$

where A_0 and A_f are respectively initial and final cross-sectional areas of failed component. Using ImageJ® Software the fracture surfaces of Figures 5-7 are analyzed to obtain changes in cross-sectional areas as a function of ageing times and environments. The calculation operations must be carried meticulously. Figure 8 summarizes the findings when equation (A) is applied for a 7 months ageing period.

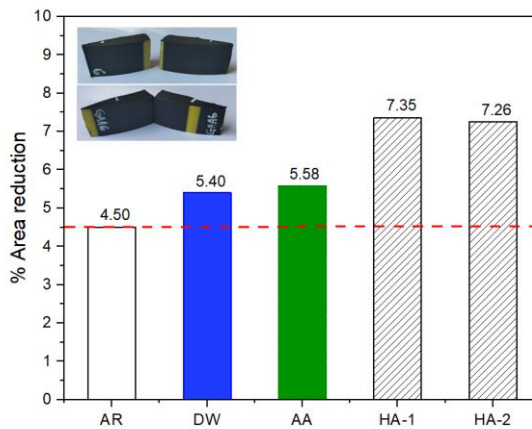


Fig. 8 Percent ductility for unaged (AR) and aged HDPE-100 in DW, AA and HA (7 months ageing period)

The first indication is that ductility of aged HDPE in HA rose by 39% compared to as-received (AR) specimens. This is an important information about the role of hydrochloric acid in increasing solubility and plasticization of semi-crystalline polymers such as polyethylene [28,33,34]. Secondly, for acetic acid (AA) ageing, ductility rise is limited only to 19% since AA is ranked as a weak acid and its behavior in this case is close to that of deionized water (DW) with 17%.

Literature does not provide impact ductility data for aged arc-shaped HDPE pipes. However, when considering filaments machined from HDPE pipes and aged in aggressive environments for 3 months [21], it is possible to discuss ductility in terms of strain at failure (ϵ_f). The latter property is dependent on the strain-hardening capacity of the material. It is found that ductility is increased to 900% (H₂SO₄), 1400% (crude oil) and 850% (Toluene-Methanol mixture) as compared to 700% for As-Received (AR) specimens. The rank according decreasing strain at failure is as follows [21]:

$$\epsilon_f (\text{crude oil}) > \epsilon_f (\text{Toluene-Methanol}) > \epsilon_f (\text{H}_2\text{SO}_4) > \epsilon_f (\text{AR}) \quad (7)$$

It is concluded that both methods (%Ar and ϵ_f) agree that aggressive environments augment ductility because of triggering degradation processes such as solubilization and plasticization of HDPE [28,33,34,36]. It is accepted that %Ar and ϵ_f measure different types of material behavior. Strain at failure is mostly affected by uniform elongation as it depends on strain hardening while %Ar is ultimately a measure of the deformation required to produce fracture and initiate the material necking propagation [43].

4. Conclusion

This work is proposed to investigate a new adaptation for non-standard specimens to study at the same time sorption and mechanical resilience properties of HDPE gas pipes after long-term chemical ageing. The following conclusions are made:

- For reasons related to the preservation of the thermomechanical history imparted by the extrusion process of HDPE-100 pipes and by handling/storage methods, the choice of non-standard specimens might be favored in order to implement, much more representative environmental stress cracking experimental studies and to extend actual testing standards.
- The designated procedure allowed obtaining necessary information to characterize sorption of weak and strong acids. Within 2 weeks, HDPE-100 resin experienced maximum swelling of HCl (20%). Then, a nearly total desorption took place progressively during the next 28 weeks.
- According to Weitsman's sorption labeling, deionized water, acetic and hydrochloric acids in contact with HDPE-100 resin are respectively Type (B), Type LF and Type (D). It is recognized that water sorption curve symbolizes the condition of a two-stage diffusion. For the strong acid, the behavior of sorption/desorption is in agreement with chemical and physical degradation of the resin and antioxidant depletion.
- Deionized water experienced a slow and monotonic decrease of Charpy energy which can be attributed according to literature to the absence of plasticized material resulting in and enhanced swelling. For the 7 months ageing period, both acids show much higher Charpy energies than as-received and deionized water cases. For HCl (20%), the increase is 110% and for CH₃COOH (5%), it is 75% compared to as-received Charpy energy, while deionize water experienced a 25.8% decrease. It implies that HCl and CH₃COOH made the HDPE resin more resilient but brittle as inferred from fractography and literature.
- Fractographic analyzes show that crack initiation is driven by a craze zone, the size of which corresponds to b_1 distance worked out from post-failure surface observation. As expected, crack propagation is completed by a crack arrest line defining the y_1 parameter. Side thinning (or "shear lips") and craze zone size (b_1 parameter) are amplified for acids while the distance to final yielding (y_1 parameter) remained almost constant for all environments.
- The results suggest that the various deformations incurred by aged material represent a global and realistic effect of chemical and mechanical processes induced over time. It

is believed that such combination can serve for a more representative studies of durability/reliability for HDPE pipes in interaction with service environments. The prospects will still open with the provision of other characterizing measurements related to ESC phenomenon.

- Ductility is approached using % cross-sectional area reduction at break. For HA and AA ageing, it increased respectively by 39% and 19% compared to as-received specimens. This is another good indication about hydrochloric acid contribution into solubility and plasticization of HDPE. This finding is corroborated from literature using strain at failure (ϵ_f) as a measure of ductility for chemically aged HDPE filaments.

Abbreviations

| | |
|----------------|--|
| AA | Acetic acid; |
| A ₀ | Initial cross-sectional area (%); |
| A _f | Final cross-sectional area (mm ²); |
| Ar | Area reduction (mm ²); |
| AR | As-received; |
| CE | Charpy energy; |
| DW | Deionized water; |
| ESC | Environmental stress cracking; |
| ESCR | Environmental stress cracking resistance; |
| GA | Gaz pipe, As-received; |
| HA | Hydrochloric acid; |
| HDPE | High-density polyethylene; |
| LF | Linear Fickian; |
| MDPE | Medium density polyethylene; |
| MP | Micro plastics; |
| OD | Outer diameter; |
| PE | Polyethylene; |
| PP | Polypropylene; |
| RCP | Rapid crack propagation; |
| SCG | Slow crack growth; |
| SDR | Standard dimension ratio; |

Nomenclature:

| | |
|-----------------|---|
| b ₁ | Extent initiation jump (mm); |
| b ₂ | Extent propagation jump (mm); |
| δ | Solubility parameter (MPa) ^{1/2} |
| ϵ_f | strain at failure (%); |
| G _{IC} | Elastic fracture toughness (kJ/m ²); |
| J _{IC} | Elasto-plastic fracture toughness (kJ/m ²); |
| m | Mass (g); |
| m ₀ | Initial mass (g); |
| OIT | Oxidation induction time (min.); |
| t | Time (hours or days); |
| t ₀ | Initial thickness (mm); |
| y ₁ | Distance to final crack arrest line (mm); |
| Δt | Thickness change (mm); |

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