STUDY OF CRUDE OIL INTERACTION WITH HIGH DENSITY POLYETHYLENE PIPE SURFACE MATERIAL

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Abstract:

Polymeric materials are more and more solicited for industrial applications than metals essentially for acceptable strength, durability, maintenance techniques and cost reasons. However, both resistance to degradation and maintenance issues, forced to establish high performance polyethylene (PE) resins to be used in many aggressive environments. The environmental stress cracking (ESC) phenomenon occurs when PE is subjected to stresses in the presence of unavoidable adverse chemical conditions. Parameters such as time-to-failure and long-term brittle-like failure of PE pipes present several similarities in relation to ESC and slow crack growth (SCG) phenomenon. This study presents some results of the interaction between crude oil (CO) and high density polyethylene (HDPE) pipe surfaces (outer and inner layers). It is found that more liquid is absorbed by the outer pipe surface than the inner one. After 7 days exposure to CO, the elasticity modulus is reduced by more than 76%. While most of the mechanical properties have been negatively affected, it is observed that the breaking stress (σ_f) increased by 2.5 and 1.6 times respectively for inner and outer layers compared to the as-received case. In addition, it is noted that roughness (R_a) increased by ~48% and varied a little for outer and inner surfaces respectively. Such observations are poles apart compared to previous studies using toluene-methanol environment. Structure analysis in terms of crystallinity, oxidation induction time and morphology observations are used to formulate a rationalization for these results and the differences with other solvents.

Keywords: Polyethylene pipe, Crude oil, Environmental stress cracking, Degradation, Mechanical properties, Crystallinity, Roughness, Hardness.

1. Introduction

The use of HDPE pipe in oil and gas industry is dictated by the suitable chemical properties of polyethylene. Unlike steel and glass-reinforced epoxy pipes, polyethylene pipes are resistant to corrosion, swelling and chemical degradation at moderate temperatures. For longer service periods, the initially designed pressure capabilities of HDPE pipes are mostly preserved. For instance, according to a report issued by the Plastics Pipe Institute, the use of reinforced thermoplastic pipes (RTP-HDPE) allowed saving up to 50% on installation costs and required time in comparison with steel pipes [1]. In another case, when checking the remaining properties of PE-100 pipe oil flow lines after 14 years (168 months) operation in the State of Oman, it was found that σ_y (max. stress) exceeded 25 MPa and ε_f was in the range 380% to 427% (>350%). These two properties are in satisfactory limits with the mandatory specification. The properties are test out at 3 locations across the wall (internal, middle and external layers). In addition, the OIT is measured at five points across the wall thickness of the pipe and the lowest reported level was 43 minutes which much higher than the specified minimum (OIT > 20 min.) [2].

Extrusion is the main process for the manufacturing of HDPE pipes. It is known that this process is responsible for a multitude of morphological and intrinsic heterogeneities through the pipe wall. Subsequently, pipe service behavior is greatly influenced by embedded morphological and internal stress characteristics [3,4]. During extrusion, HDPE pipes are directly showered with water baths, provoking an immediate quenching of the outer layer (OL) while the inner layer (IL) undergoes slow cooling in free circulating air. Such temperature gradient creates a distinctive distribution of internal stresses in pipe wall: (i) compressive and (ii) tensile stresses respectively at OL and IL.

Such stress state is reinforced by the geometrical form which holds the product from any important later deformation. Consequently, it is legitimate to assert that eventual crack propagation from outer flaws will be prevented because of reduced crack-tip stresses in a compressive dominant stress field. Slow cooling of the inner surface usually facilitates the rearrangement of the macromolecules in a semi-crystalline structure which enhances its crystallinity. Nevertheless, these cooling conditions facilitate reducing the level of initially incorporated antioxidants as they migrate to the surface and finally will be consumed by the surrounding oxygen [5]. On the other hand, the rapid cooling of the outer pipe surface decreases the local crystallinity while freezing the antioxidants in the resin. That is why pipe structural characterization usually shows more antioxidants in the outer layers compared to the inner ones [6].

The other most important constraint when using polyethylene pipes networks in distribute or transport hydrocarbons remains the joint interaction between the PE and surrounding fluids. In general, this situation evolves towards environmental stress cracking degradations in the form of swelling, fluid contamination, losses across pipe wall, surface cracks and changes in mechanical and chemical properties. For the case of some organic liquids sorption, Table 1 summarizes some published results for the 3 main technical applications of HDPE as pipes, films or geomembranes.

MATERIAL/ PRODUCT	EXPERIMENTAL CONDITIONS	HYDROCARBON		SATURATIO N TIME (h)	GAIN (%)	REF.		
Geomembranes; HDPE		Benzene		42	0.12	7		
test sheets; 350x300x1.6	Sorption and desorption;	Toluene		37	0.11			
mm^3 .	Gravimetric method, 25°C	Acetone		106.7	0.02			
HDPE film "cast";	Permeability study,	Toluene/Methanol		Tol: 50 min.	High	8		
0.4 mm thick	Chromatography, 60°C, (Test. time: 140 min.)	TM mixture 50:50		TM: 48 min.	Low	8		
HDPE compression molded sheets; (30x6x1.18 mm ³)	Raman spectroscopy, 30°C (Relaxation: 18h)	Crude oil (mixture)		3	6.9	9		
HDPE;		Toluene (pol	ar)	8.6	-			
Pre-pressed sheets; 305x305x1.65 mm ³ .		1-butanol (non polar)		1324.6	-	10		
HDPE pipe SDR 9,	Gravimetric method. (2500 h)	Toluene		16.2	-	-		
2.61 mm thick.		1-butanol		966.7	-			
HDPE pipe; Machined	Ageing, Methods: FTIR,	Dissel all (New	50°C	336	8.4			
sheets; 80x10x2mm ³ .	TGA, (Test. time: 145 days)	Diesel oil (Non- polar)	20°C	1080	6.7	11		
PE pipe grade, samples: 50x20mm ² ;	Solubility, diffusivity, Weight	Methyl ester (Biodiesel, Low	23°C	173.6	9.5	- 12		
0.6 mm thick.	loss method.	polarity)	70°C	1.2	4.7			
		Toluene		168	0.078			
HDPE pipe;	Sorption and desorption; 23°C	1-Butanol		>4 months	< 0.005	12		
2.59 mm thick,	Sorption and desorption, 25 C	Crude MCHM				- 13		
Monomodal.	MCHM : [(4-methylcyclohexyl)-Methano		1]	>4 months	< 0.005			
PE pipes; branched, 1.8 mm thick, χ _c =58%, diameter 12 mm.	Sorption, sample of 10 mg, (Test time up to 541 days)	Internal: liquefied propane and external: air gaseous propane		400	3.8	14		

Table 1. Typical published saturation (equilibrium) times for a variety of hydrocarbons in contact with
PE materials.

For crude oil in contact with compression molded PE sheets, within a period of 3 hours, the % weight gain is relatively high (\sim 7%) [9]. However, when considering petroleum components such toluene the absorbed amount remains low. From literature, it is expected, that temperature usually intensifies the sorption phenomenon of the liquids; this is due to the increase of PE free volume that allows more permeation of organic molecules.

In this work, the interaction of crude oil with both internal and external surfaces of a PE pipe is studied. The results are considered in terms of mechanical and surface quality properties in relation to eventual material structural alterations.

2. Materials and experimental methods

2.1. Specimen preparation

The piping material used in this study is extruded from HDPE resin (HOSTALEN CRP 100 black). It is a PE100 grade with 200 mm outside diameter and 11.4 mm thick. It is manufactured STPM CHIALI Co (Algeria). Its density is 0.959 g/m³ and its melt flow rate is 0.23 g/10min. The measured elastic limit (σ_y) is ~23 MPa and its Young's modulus averages ~1100 MPa. The oxidation induction time (OIT) is > 30 minutes implying an excellent resistance to stress cracking. Envelopes 2 to 4 mm thick and 270 mm long are machined according to a specific experimental protocol [15] to represent

the most exposed layers of the tube (internal and external). Figure 1 illustrates intermediate phases and specimen geometries prepared according to ASTM recommendations for roughness and hardness measurements (Fig. 1a), tensile tests (Fig. 1b) and sorption tests (Fig. 1c).

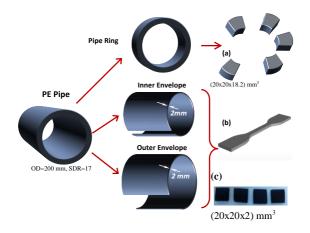


Figure 1. Machined specimen geometries for: (a) Roughness and hardness measurements; (b) Tensile tests and (c) Sorption test.

2.2. Characterization techniques

HDPE samples weighing is performed using an electronic balance Type ABS 220-4S from KERN & Sohl GmbH with a limit of 220 g and a precision of 0.1 mg. As-received specimens are conditioned in dry tight plastic containers until measuring time while ESCR specimens are immersed in Algerian crude oil (Sahara Blends) solution using glass jars with tight covers. Crude oil is provided by the National Oil Company (SONTRACH) from the refining facility of Skikda. The mechanical behavior is assessed by tensile tests using a Zwick 1120 testing machine. A drawing speed of 50 mm/min is used. To study the topography of the internal and external surfaces of the pipe, an Altisurf 500 3D profilometer is used. Data acquisition and processing of standard roughness criteria (R_a , R_q and R_z) is performed by Altimap software, according to ISO 4287 (eq.1a, b and c).

$$R_a = \frac{1}{N} \sum_{i=1}^{N} |Y_i| \tag{1a}$$

$$R_q = \sqrt{\frac{1}{N}\sum_{i=1}^{N}Y_i^2}$$
(1b)

$$R_Z = \frac{1}{5} \sum_{i=1}^5 Y_{p_i} + \frac{1}{5} \sum_{i=1}^5 Y_{\nu_i}$$
(1c)

where *N* is the number of events (depth or peak); Y_i are the deviations from a mean line; Y_{pi} is the highest profile peak and Y_{vi} is the depth of the lowest profile. Crystallinity measurements are performed with a Mettler TA 3000 DSC with a heat rate scan of 10°C/min. Crystallinity is calculated as the ratio of the changes in melting enthalpies of the pipe material to the perfectly crystalline PE according to equation 2:

$$\chi_c = \frac{\Delta H_f}{\Delta H_{f_0}} \times 100 \tag{2}$$

where ΔH_f and ΔH_{f0} in (J/g) are respectively the changes in melting enthalpies of the pipe material and the perfectly crystalline polyethylene. The reference value for ΔH_{f0} is usually taken from literature as 293 J/g. The Shore D hardness measurements are performed using a portable durometer Mitutoyo Hardmatic (HH-401). Usually, 5 samples per surface case are considered and 6 data points are extracted per sample surface for exposed and unexposed surfaces. The *OIT* analysis is carried out using a 200 PC NETZSCH according to the EN 728 AFNOR standard at 200°C. The OIT then corresponds to the time required for the disappearance of all the stabilizing functions.

3. Results and discussion

3.1. Percent Weight change

The experimental sorption data for both IL and OL in CO are presented in Figure 2. The outer surface absorbs more liquid than the inner one which can be explained by the usually amorphous structure of OL following the quenching operation during pipe extrusion. In other words, it is the crystallinity that assumes the role of barrier during the immersion of the polymer in liquid media.

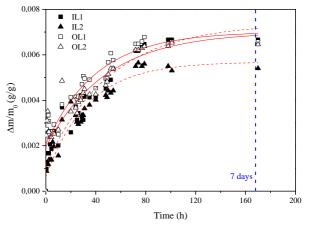


Figure 2. Evolution of % mass change in CO environment for inner (IL1 and IL2) and outer (OL1 and OL2) HDPE pipe layers as a function immersion time.

It is observed that the sorption of CO in HDPE follows a very slow process, with a percent mass gain that does not exceed 0.7% in 70 hours. The data show some scatter since the method involves removing specimens from the environment, paper drying and weighing in a very short time [7,10,12-14]. The fitted correlations for the 4 experiments (inner: IL1, IL2; and outer: OL1, OL2) are obtained using the following equation as a function time (hours):

$$\frac{\Delta m}{m_0} = a + b e^{-cTime} \tag{3}$$

The calculated coefficients (a, b and c) for equation (3) and the corresponding standard errors are summarized in Table 2. It is found that the exponent model fits adequately the experimental data. The maximum standard error is around 1.5 % and the determination coefficients are acceptable although the experimental process for each obtained data point is critical (sample handling, drying, and weighting methods). It is noticed that the obtained data lay in the large spectrum shown in Table 1 even if saturation is not considered yet well established. It is believed that such process can take longer

times as in the case of monomodal HDPE pipe (Table 1) [13]. In literature, it is found that small size pipes are more vulnerable to permeation than large size pipes. Pipes with water stagnation periods create a much higher risk of exceeding the maximum contaminant level of benzene than pipes with continuous water flow. Additionally, a PE pipe buried in soil of elevated organic matter is proven to permeate to a less important extent than a pipe buried in a soil of lower organic matter [16].

_ Coefficient Standard							
Layer	(Equation 3)		Error	R ²			
IL 1	a	0.00739	5.04E-04				
t= 2.3 A=22.56x20.89	b	-0.00626	4.81E-04	0.928			
$m_0 = 1.0202$	с	-0.01954	0.0033				
IL 2 t= 2.55	а	0.00569	2.86E-04				
A=22.76x19.82	b	-0.00504	2.89E-04	0.931			
$m_0 = 1.0179$	с	-0.02982	0.00438				
OL 1	a	0.00701	5.28E-04				
t= 2.48 A=20.12x20.44	b	-0.00495	5.16E-04	0.834			
$m_0 = 0.9879$	с	-0.02468	0.00617				
OL 2	a	0.00701	6.42E-04				
t= 2.9 A=21.04x21.01	b	-0.00506	6.15E-04	0.824			
$m_0 = 1.2240$	c	-0.02065	0.00563				
t: thickness, mm, A : cross sectional area, mm^2 , m_0 : mass, g.							

 Table 2. Fitted correlations for sorption tests.

3.2. Mechanical behavior

The mechanical behaviors for the internal and external layers are shown in Figure 2. The overall shape of the curves for as-received and CO cases is typical of a semi-crystalline polymer characterized by a reduced elastic domain, followed by a yielding zone identifying the maximum stress and the beginning of the plastic flow. The global comparison between both cases indicates that many changes occurred after exposure to CO environment; higher yield stress and higher failure strains are recorded. This suggests that CO is acting as an agent favoring material ductility.

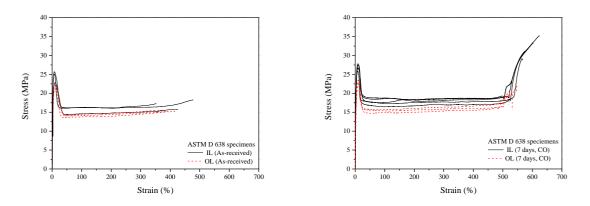


Figure 2. Engineering stress strain curves of inner and outer pipe layers for the as-received (AR) and crude oil (CO) cases.

For the IL (as-received), a narrow elastic domain is revealed for a deformation interval (0-3.7%), followed by an inelastic zone up to 8%. This is the domain of homogeneous deformation before the yielding which is associated with the threshold plastic flow and the appearance of the necking phenomenon. In the deformation range of (26-420%), the material flow settles where the necking will propagate along the specimen; then, within the range 420-480%, the hardening phenomenon is initialized once the necking has ended. At this stage, the deformation process becomes homogeneous and the mechanism separation turns to a fibrillar morphology. Finally, failure (final separation) takes place at around 480%. Similar analysis for OL shows a slight shift in deformation with respect to IL, i.e., the elastic domain extends up to 4% deformation while material flow starts at 9%. The cold drawing zone lies between 37% and 400% strain and ultimate breaking occurred at 470%. The effect of CO is markedly illustrated for IL. The elastic zone is narrowed when yielding took place within the range 8% - 25%. Plastic flow extended from 25% to 520% strain. Also, the effect of CO is striking on the OL as the elastic zone went up to 5% and initial necking started around 34%. Plastic drawing extended until 500%. It is deduced that CO enhances larger cold drawing followed by extensive hardening for both IL and OL layers. Some enhanced mechanical properties have reported in literature after contact with particular chemical environments [2, 17-19].

Measured mechanical properties are revealed in Table 2. Generally, it is observed that the initial pipe IL is stronger than the OL in terms of *E* and σ_y . In terms of deformations, it is rather the OL which occupies a better position. Once IL is exposed to CO, *E* is reduced by roughly 76%. For the OL, the variations are much smaller (~15%). The stress at failure (σ_f) increased respectively for IL and OL by 2.5 and 1.6 times. This means that CO hardens PE material and allowing more or less ductility.

	Pipe Layer	E (MPa)	σ _y (MPa)	σ _f (MPa)	<i>E</i> f (%)	Ref.	
As- received	п	1725.72 (±93.25)	25.28 (±0.26)	17.15 (±0.38)	441.16 (±59.87)	1)	
	IL ·	723.20	23.13	29.85	506.04	2)	
		871.30	22.53	15.55	413.83	1)	
	OL	(±43.40)	(±0.21)	(±0.38)	(±13.26)	· · ·	
		423.46	18.24	25.92	425.30	2)	
7 days immersion in CO	IL	253.00	26,88	30,37	579,50		
		(±88.00)	(±0,59)	(±3,35)	(±26,65)		
	OL	642.66	22.89	20.72	531.71	1)	
		(±40.44)	(±0.38)	(±0.89)	(±9.87)		
14 years oil transmission in (Sahara of Oman)	IL	847	26.0	18.0	426		
	OL	865	25.7	14.2	379	3)	

Table 2. Comparison of some mechanical properties for IL and OL after exposure to CO environment.

¹⁾ Present study; ²⁾ [17]; ³⁾ [2].

3.3. Surface–-Structure properties

The obtained measurements for roughness criteria (R_{α} , R_q and R_z) together with other properties (HS, χ_c and OIT) are shown in Table 3. In the case of CO, R_a increased by 48% for the outer surface and varied little for the inner surface. Such increase usually indicates that surface alterations or extracted species are polluting the immersion environment. It should be mentioned that the outer surface is subjected to the extrusion die pressure which can leave furrow-like imprints contributing to increased roughness.

Surface Side	Environment	Roughness (µm)			Shore Xc	χc	OIT
		R_a	R_q	R_z	Hardness	(%)	(min.)
Inner	As-received	0.948	1.127	4.152	48.90	64.26	22.7
	Crude Oil	0.926	1.077	3.423	39.65	45.18	14.0
Outer	As-received	0.837	1.034	3.644	60.60	47.79	26.5
	Crude Oil	1.253	1.457	5.055	47.90	40.54	21.4

Table 3. Surface and structure properties of inner and outer surface layers.

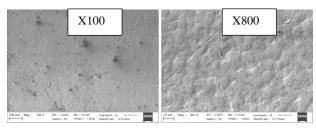
The Shore hardness of both surfaces is also depicted in Table 3. It indicates that the outer surface is harder than the inner surface despite a better crystallinity of the latter. The lamellar flow during extrusion usually induces such variation in the pipe structure.

After immersion in CO, the hardness of both surfaces decreased: -18% and -20% for inner and outer surfaces respectively. This decrease can be attributed to the degradation of the structure underneath surface layers. The decrease in the degree of crystallinity generally is among the causes of hardness degradation [6]. Usually, for HDPE ambient air ageing, crystallinity increases because of recrystallization process. However, the inverse situation can occur due to the surrounding conditions, i.e. the ever presence of oxygen, which might react with the polymer causing crystallinity decrease [20]. In other studies, it is suggested that a crosslinked structure takes place and would not allow large chain rearrangement, which ultimately reduce the mobility the chains and at the same time, it prevents polymer chains to re-align [6, 21].

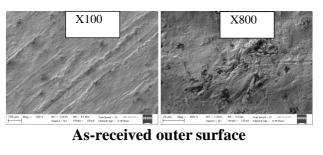
In order to minimize oxidation reactions under the effect of aggressive environments, manufacturers add additives such as antioxidants (AO) and carbon black (CB) to the resin. During pipe extrusion, a quantity of OA is consumed and another one remains in the resin; the latter provides a long-term protection for the pipe. Originally, the OIT parameter is 30 minutes, but after extrusion it is only 26.5 min for the outer surface and 22.7 min for the inner surface (Tab. 3). Antioxidants remain frozen in the resin due to quenching of the cooling water on the outer surface. In contrast, the internal surface cools slowly by convection and the reduction of AO concentration takes place by migration to the surface and subsequently their consumption by the surrounding oxygen. This induces a gap of 14% between OIT values at external and internal layers. This difference becomes 34% once exposed at CO for 7 days. It is suggested that CB and other additives are dissolved in the immersion liquid. To test this hypothesis, microscopic observations are performed as shown in figure 3.

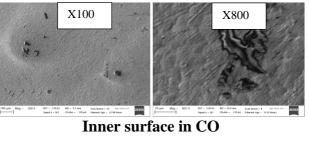
It is clear, for the as-received case, that external and internal surfaces do not have the same morphology. The inner surface seems smooth with spherulites of diameter around 10 μ m, while for the external surface remains rough with imprints probably coming from the extrusion operation.

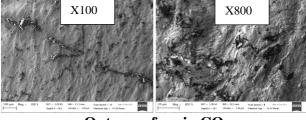
According to literature, the spherulitic structure is not observable on the external surface [22,23]. The presence of agglomerated species is noted on both surfaces before and after immersion in solvents with a noticeable increase for the external surface. This makes it possible to conclude that the morphology of the external surface seems very affected by the contact with these types of organic media [24,25]. This study is important for the PE pipe manufacturing industry as it opens new applications for transporting hydrocarbon components in plastic pipes probably with lower costs and more efficiency in pipe replacement. Also, this study should contribute in enhancing usage of PE materials for automobile fuel systems.



As-received inner surface







Outer surface in CO

Figure 3. SEM observations at inner and outer surfaces, before and after immersion in CO at 100X and 800X.

4. Conclusion

This preliminary work presents the effect of Algerian crude oil on mechanical and surface properties of the HDPE pipes. It is presented as effects on outer and inner surface layers. The following main conclusions are drawn:

- 1. The outer layer absorbs more crude oil (CO) than the inner layer. This can be explained by the amorphous morphology of extruded HDPE copolymer that predominates towards the outer pipe layers.
- 2. Tensile tests show that the as-received inner layer (IL) is more resistant than the OL in terms of Young modulus and elastic limit, whereas in terms of deformations, it is rather the outer layer that becomes the most tenacious.
- 3. After 7 days exposure to CO, the elastic modulus is reduced by more than 76%. Most of

mechanical properties decreased but in the case of stress at break; it is surprising increased by 2.5 and 1.6 times respectively for the IL and OL. This could be related to increased material ductility which is confirmed from strain at break values.

- 4. The CO effect resulted in a decrease of roughness for IL and inversely increased that of the OL. The explanation is related to migration and loss of anti-oxidants at the outer surface. The carbon black additives may play another role in roughing the surface especially during extrusion process.
- 5. The outer surface remains harder than the inner one despite a better crystallinity of the latter which is confirmed from literature.

ABBREVIATIONS:

- AR : as-received
- CO : crude oil
- ESC : environmental stress cracking HDPE: high-density polyethylene
- HS : Shore hardness
- *IL* : inner pipe layer
- *OL* : *outer pipe layer*
- PE : polyethylene

NOMENCLATURE:

- E : Young's modulus, MPa
- $\Delta m/m$: % mass absorbed, g/g
- *OIT : oxidation induction time, min.*
- R_a : arithmetical mean roughness, μm
- R_q : root mean square roughness, μm
- R_z : average maximum height of the profile, μm
- χ_c : crystallinity, %
- ε_f : failure strain, %
- ε_{v} : yield strain, %
- σ_f : failure stress, MPa
- σ_y : yield stress, MPa

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