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Influence of accelerated ageing (Temperature and acid attack) on mechanical properties, structural and morphological characteristics of high-density polyethylene sewer pipes

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ABSTRACT

This article analyzes the aging effect of HDPE sewer pipes when exposed to sulfuric acid at different temperatures. The mechanical properties, structural and morphological changes were evaluated. Results showed an impressive environmental degradation of HDPE. The tensile test showed that with a well-correlated increase in immersion time and higher temperatures, the elongation and ultimate stress of HDPE decreased. At 60 °C, the elongation at break indicated, at every time interval, an abrupt drop and a simultaneous loss of ductility. The micro-structural FTIR absorptions at 719–730, 1462–1472, 2848, 2916 and 3400 cm⁻¹ were detected and compared using the spectrum of an unaged HDPE. The modification of the mechanical properties seems to correlate well with the structural observations obtained from the scanning electron micrographs. Eventually, it was discovered, via thermal aging studies, that the combination of temperature and sulfuric acid appears to be a major factor of high-density polyethylene breakdown.

Keywords: Deterioration, HDPE, mechanical properties, sewers, sulfuric acid

1. INTRODUCTION

Plastic pipes have become gradually important throughout the last years. Their lifetime and the factors that might influence this lifetime are highly discussed among experts. Plastic tubing has been used in many different ways in the private as well as in the industrial sector: industrial plants, sanitary installations, biogas plants, irrigation systems, food industry, chemical process industry and as sewer pipes in the field wastewater [1]. These pipes are mainly used because of their resistance to corrosion. Recently, the polyethylene dependent material has become more and more popular in the piping industry. Indeed, those materials are an economical, superior performance and durable solution. Nevertheless, using them in harsh environments such as marine environment, sulfate environment, UV exposure, humidity, wide temperature range, etc... can affect their lifetime and degrade their mechanical and physical characteristics [2].

The study of natural aging takes time. With a view to comprehend and foresee the HDPE behavior at various maturities but in a short period of time, it's necessary to use accelerated aging. An accelerated aging test is used to simulate real-time shelf life aging. It allows us not only to

control the similarities of the materials behavior with natural aging, but also extrapolations.

Using saline solution, Guermazi et al. [3] investigated how temperature and aging time affected mechanical and structural qualities of pipe lining. They discovered that high temperatures can lead to severe mechanical deterioration of the PE coating.

Salem et al and liu et al. [4,5] carried out a study to determine the effect of hygrothermal aging on the tribological behaviour of high-density polyethylene (HDPE) and the impact resistance of carbon fiber reinforced epoxy polymer (CFRP). The results showed that the aging performed affected the surfaces of the materials (degradation phenomenon of the HDPE and the decrease in the shock resistance of the CFRP).

Other interesting research results came from the studies of Guermazi et al. [6] in their experiments with a pipe lining utilizing 2 solutions: distilled water and synthetic sea water. Both solutions were used at 70°C steady temperature and multiple aging cycles.

El brahmi et al. [7] carried out a study on wastewater in order to assess the existence of aqueous sulphides and to quantify the influence of wastewater quality parameters on their concentration. The latter is formulated to the existing Sulphur cycle in wastewater. Sulphate-reducing bacteria (SRB) dominate the biological reduction process under anaerobic conditions (oxygen deficiency $<1\text{mg/l}$) and temperature between 15 and 20°C [8]. The results obtained showed that the wastewater quality parameters (Biological Oxygen Demand in 5 and temperature) influence the accumulation of sulphides.

Foorginezhad et al. [9] examined the effects of moisture, pH, H_2S and temperature on the deterioration of wastewater pipes of various types (cement, plastic and metal). The results showed that each studied parameter has a direct influence on the corrosion of the pipes. They also suggested making an online monitoring of these parameters.

Nielsen et al. [10] quantified the kinetics of hydrogen sulphide adsorption and oxidation on concrete and plastic pipe surfaces (HDPE and PVC) and studied the rate of pipe aging and sulphur odour emissions. They found out that the adsorption and oxidation rate of hydrogen sulphide on pipe surfaces is higher in concrete pipes than in plastic; and the rate of Sulphur odor generation is more remarkable in HDPE and PVC pipes.

We have studied in this work the immersion aging of a HDPE pipe utilizing three characterizations to indicate the HDPE deterioration under H_2SO_4 solution at different temperatures: mechanical (by tensile testing), structural (by FTIR) and morphological (by SEM). The primary goal is to offer ratings for how long these HDPE pipes will last in a sulfate environment. The result should help us comprehend how the loss of mechanical characteristics, structural changes and morphological observations can be correlated.

2. MATERIAL AND METHODS

2.1. Materials

The properties of the HDPE (delivered from Plastima) material used in this study are:

- Density (at 25°C) = 955 kg/m^3 (ISO 1183-1),
- melt index = 1.4 g/10min (ISO 1133),
- Viscosity = $200\text{ cm}^3/\text{g}$,
- Resistance to heat (ISO 12091 oven test) to 110°C ,
- Softening temperature = 122°C ,
- Melting temperature = 133°C .

2.1.1. Fabrication of the test pieces

The inner wall of a corrugated double-wall high-density polyethylene pipe (NM EN 13476-2) with a 1000 mm diameter served as the study's standard tensile specimen

(Figure.1). The samples were obtained by transversely cutting the tube (Figure.2).



Figure 1 Standard corrugated double wall HDPE pipe.



Figure 2. Different steps of cutting for chemical degradation test.

2.1.2. Experimental conditions

Various weather conditions were simulated using a hygrothermal aging process. The immersion of HDPE in sulfate solutions in airtight containers closed was carried out over several days at different test temperatures.

Three temperatures were chosen: ambient temperature, $40\pm 2^\circ\text{C}$ and $60\pm 2^\circ\text{C}$. The sulfate solution was made with 0.05 mol/L of sulfuric acid and a pH 1 ± 0.2 [11, 12].

The specimens were taken during the test to track the progression of weight gain. Before weighing, the samples

were removed from airtight containers closed and positioned in a different water pan to rinse for one hour.

2.1.3. Test pH value of sulfuric acid solution

A digital pH meter was used to control the pH of the 0.05M sulfuric acid solution. It was adjusted daily to keep the pH between 0.98 and 1.2 using 95 % sulfuric acid. The solution was thoroughly agitated twice a day to reduce different concentrations of the acid within the solution tank.

2.2. Testing and Characterizations

2.2.1. Evaluation of mechanical properties

The mechanical properties of the specimen were obtained in tension mode using a servo-hydraulic MTS system. The sample's measurements were established in accordance with international standard NF T 54-026 [13]. All the experiments were conducted in a laboratory at ambient temperature and 60 % relative humidity at 40 mm/min crosshead speed.

Experiments were carried out in triplicate and mean values were calculated. The purpose of the tensile tests was to see if any deterioration in the mechanical properties of the immersed samples could be observed. The tensile strength, the modulus of elasticity as well as the stress and the elongation at break (or deformation) were examined.

2.2.2. Analysis by FTIR Spectroscopy

A FTIR spectroscopy investigation was carried out to assess the

submerged samples and to ascertain whether there is a change in chemical structure or not.

The FTIR instrument used was a VERTEX 70 model, BRUKER spectrophotometry. The samples were used in lozenge form. We completed 16 scans altogether between 4000 cm^{-1} and 600 cm^{-1} , each with a resolution of 4 cm^{-1} .

2.2.3. Investigation by Scanning Electronic Microscopy

The morphology of the HDPE samples was investigated by microscopic investigation. The exposed surface of the chemically tested samples was used for SEM-EDX (QUANTA 200) analysis. An accelerating voltage of 16.0 kV was used to measure the images.

3. RESULTS & DISCUSSION

3.1. Mechanical characteristics

Figure.3 displays the results of how aging affected some mechanical properties of HDPE. In our study and as shown on the graphics, each mechanical characteristic is pictured by a mean of 3 tests. Figure.3 presents the stress variation according to the deformation of the studied material tested under three temperatures.

The three schemes indicate that the characteristics of all stress-strain curves are the same. An abrupt decrease in stress near the yield point can be observed following an initial elastic phase. At this point (increased elongation without further increase of tensile strength), the local necking of the specimen started and the samples were susceptible to elongation and strain hardening until they broke their original gauge length. The elongation (strain at break) of the samples aged at 25, 40 and 60 °C is reduced

The loss of

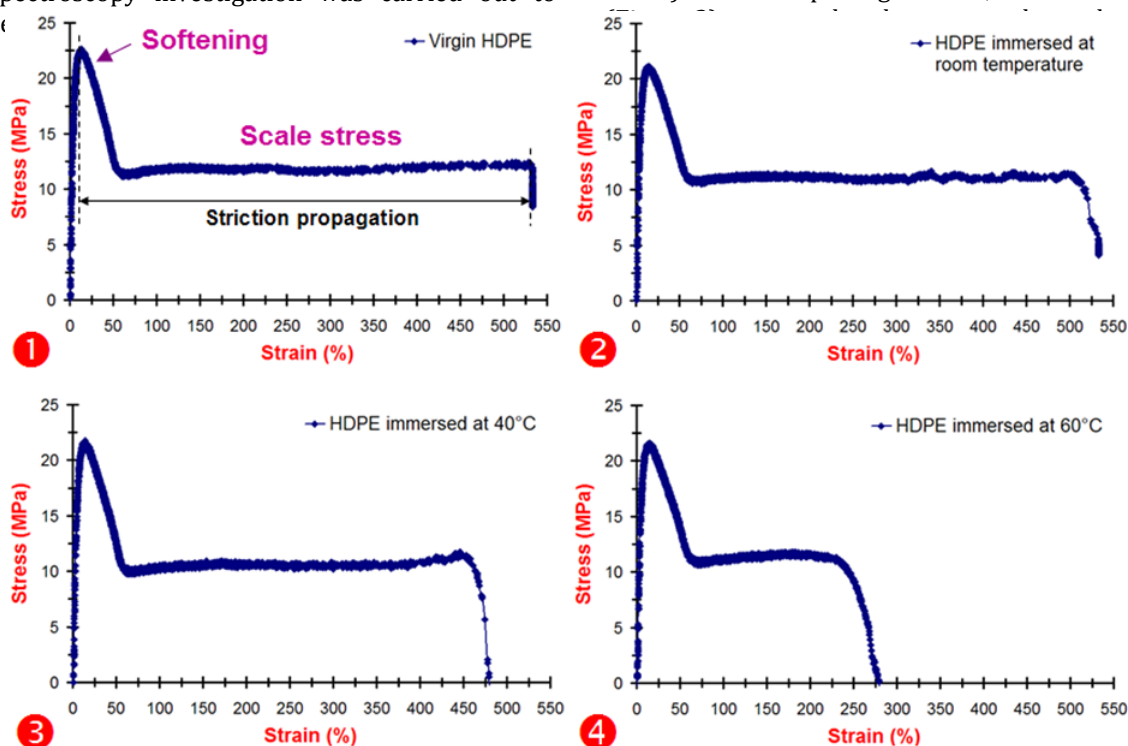


Figure 3. Effects of aging temperature on the tensile strength curve aged at 40°C (3) and 60°C (4) for 92

elastic properties increases with increasing aging temperature.

The smallest dimension at the point of fracture is influenced by the absorption of the solution: An increase of immersion time and temperature increases the thickness of the sample at the point of fracture (Figure.4 and Figure.5) after the tensile test. And it didn't change only because of the absorption of the solution before the tensile test.

Similar results has been found and depicted by microscopic images (SEM) in the literature [13,14]. The penetration of the sulfate solution into the polymer structure can explain this decline in mechanical properties. Particularly in the amorphous phase, the diffusion of sulfate solution molecules makes polymer molecular chains more flexible [15,16]. As a result, a plasticizing effect happens during the various immersion times [17]. This phenomenon has already been demonstrated in previous studies [6] by reducing the glass transition temperature T_g for samples aged by immersion. The polymer's mechanical properties deteriorate as a result of all these factors.

Similarly, it could be demonstrated that the aging temperature affects how much the mechanical properties degrade: At greater aging temperatures, the mechanical properties deteriorate more. This result might be connected to the paragraph before. The aging temperature has a considerable impact on the high-density polyethylene material's behavior during immersion: The physical and structural changes that take place during immersion can be accelerated by raising this parameter. As a result, an increase in the aging temperature gradually has a greater impact on the mechanical qualities.

The coupling impact of time and temperature was

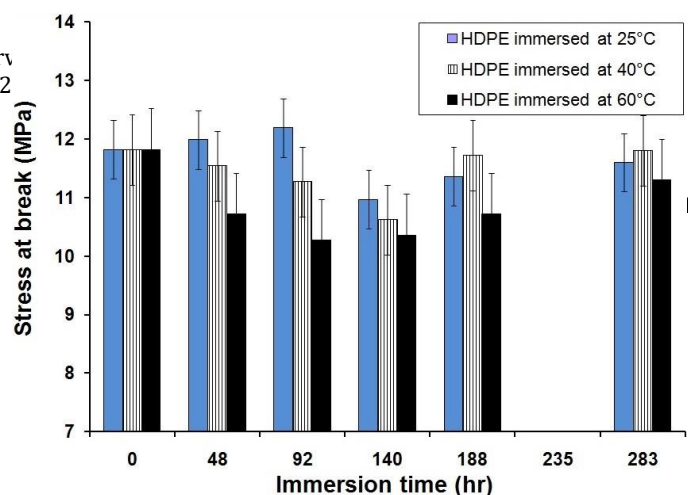
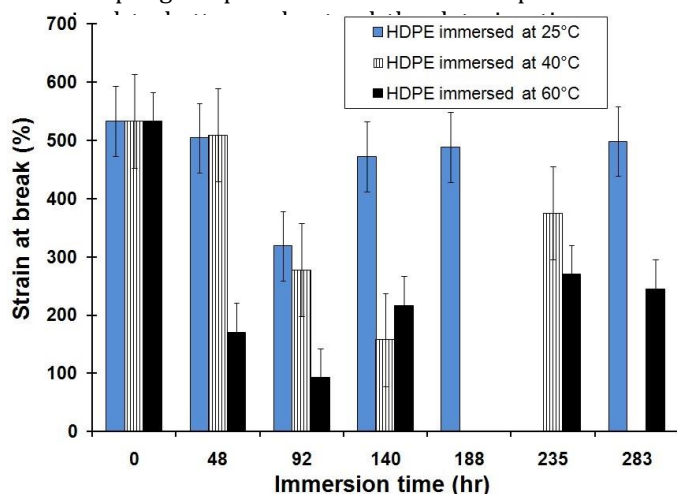


figure 5. Development of the stress at break as a function of the aging time at different temperatures.

During the experiment, we added small quantities of diluted sulphuric acid (H_2SO_4) solution to the initial solution used for immersion of the HDPE samples to maintain the pH at 1. Under the effect of aging and temperature, the elongation at break (%) decreased significantly (Figure.4) at the time of testing.

At 92 hours, we observed (according to the graphs) significant degradation for all the temperatures studied (25, 40 and 60). This degradation could be due to temperature, in addition to several factors, including: the addition of sulfuric acid to maintain a pH of 1 in the accelerated aging tank, and also to the unexpected exposure of the specimens to UV light in the room at the time of the experiment. This UV radiation can also contribute to the fragility of the samples[32]. It should also be noted that the tensile tests (at 48, 92, 140, 188, 235 and 283 hours) were performed after the end of the aging experiment (under the effect of acid and temperature), that lasted 283 hours in total (nearly twelve days).

In all cases, a clear aging effect on the elongation (or strain at break) and the stress at break, which shows a sharp drop after a few days, could be observed. However, the aging temperature continues to have an impact on this decline. Mechanical characteristics drastically deteriorate as temperature rises. At a temperature of 25°C, the elongation decreases from 525 % to 300 % within a time span of 0 to 92 hours. When the temperature is 40°C, the elongation decreases further from 525% to 150 % over a period of 0 to 140 hours. However, at 60°C, the reduction becomes more significant, with the elongation varying

from 525 % to 75 % within the time interval of 0 to 140 hours. (Figure 4)

Figure.5 depicts the stress at break. At a temperature of 25°C, the stress at break increases from 11.9 MPa to 12.2 MPa within the time range of 0 to 92 hours. Subsequently, it decreases from 12.2 MPa to 11 MPa during the period of 92 to 140 hours. When the temperature is 40°C, the stress at break decreases from 11.9 MPa to 10.5 MPa within the time span of 0 to 140 hours. However, it then increases from 10.5 MPa to 11.9 MPa between 140 and 283 hours. At 60°C, the stress at break decreases from 11.9 MPa to 10.2 MPa within the time interval of 0 to 92 hours, and subsequently increases from 10.2 MPa to 11.2 MPa from 92 to 283 hours. Therefore, the influence of temperature is readily apparent.

According to Figures 4 and 5, the variation in the percentage of (Stress at break) and (Strain at break) can be explained by the diffusion process of sulfuric acid molecules in the molecular structure of the polymer. In addition, the increase in the percentage deformation of the sample after being returned to its initial state can be attributed to the saturation (or equilibrium) state of the sample following its immersion in acid at different temperatures. Mouallif et al. have already studied the diffusion process under similar conditions [26]. These outcomes are in line with Pegoretti's findings [18], who examined how time and temperature affect the flexural strength of an aged composite.

A weakening of the hydrothermal aging can explain the experimental behavior [19, 20]. The plasticization mechanism set off by the sulfate solution molecules diffusing into the polyethylene's amorphous phase may be the culprit for this degradation.

The work of Guermazi et al. [3, 6] was based on loss of modulus of elasticity, tensile strength and 500 % strain during immersion time. Our investigations on the other hand were based on stress loss and elongation at break. A loss of elasticity over time and influences of temperature have been proven. This loss of elasticity is confirmed by the scanning electron microscope (SEM) results presented in the last part of this article.

3.2. Structural characteristics

An absorption-mode FTIR analysis was used to study the structural characteristics of the aged material in order to further explain the previously discovered mechanical deterioration [1, 6].

The following Figure.6 shows the spectra of the aged and unaged samples. The aged specimen was submerged at

three different temperatures (25, 40, and 60 °C) as was previously mentioned. The infrared spectra clearly show whether there is an age-related structural change or not. Figure.6 displays the outcomes for the spectra of the

various specimens exposed to the three temperatures for 1080 hours as well as the spectrum of the high-density polyethylene specimen that was left untreated. This graph makes the impact of immersion temperature, which will be covered later, very evident.

Table 1 displays the most significant band under investigation, its functional group, and the pertinent absorption type. Table 2 displays the behavior of each of the chosen bands at the three aging temperatures. The type of development followed by individual band patterns indicates whether they are growing, expanding, or staying the same. According to the analyzed IR bands, Table 3 displays the evolution of numerous structural modifications and chemical changes as a function of aging temperature.

The results show that structural changes occur after 1080 hours of immersion, as shown by the increase in intensity of the groups (719, 1117 and 3400 cm^{-1}), by the decrease of the intensity of the groups (730 and 2848 cm^{-1}) compared to virgin HDPE and the creation of ketone and ester groups by oxidation (1368, 1738 cm^{-1} respectively). This oxidation phenomenon of polyethylene molecules could lead to the start of polymeric chain scission [21].

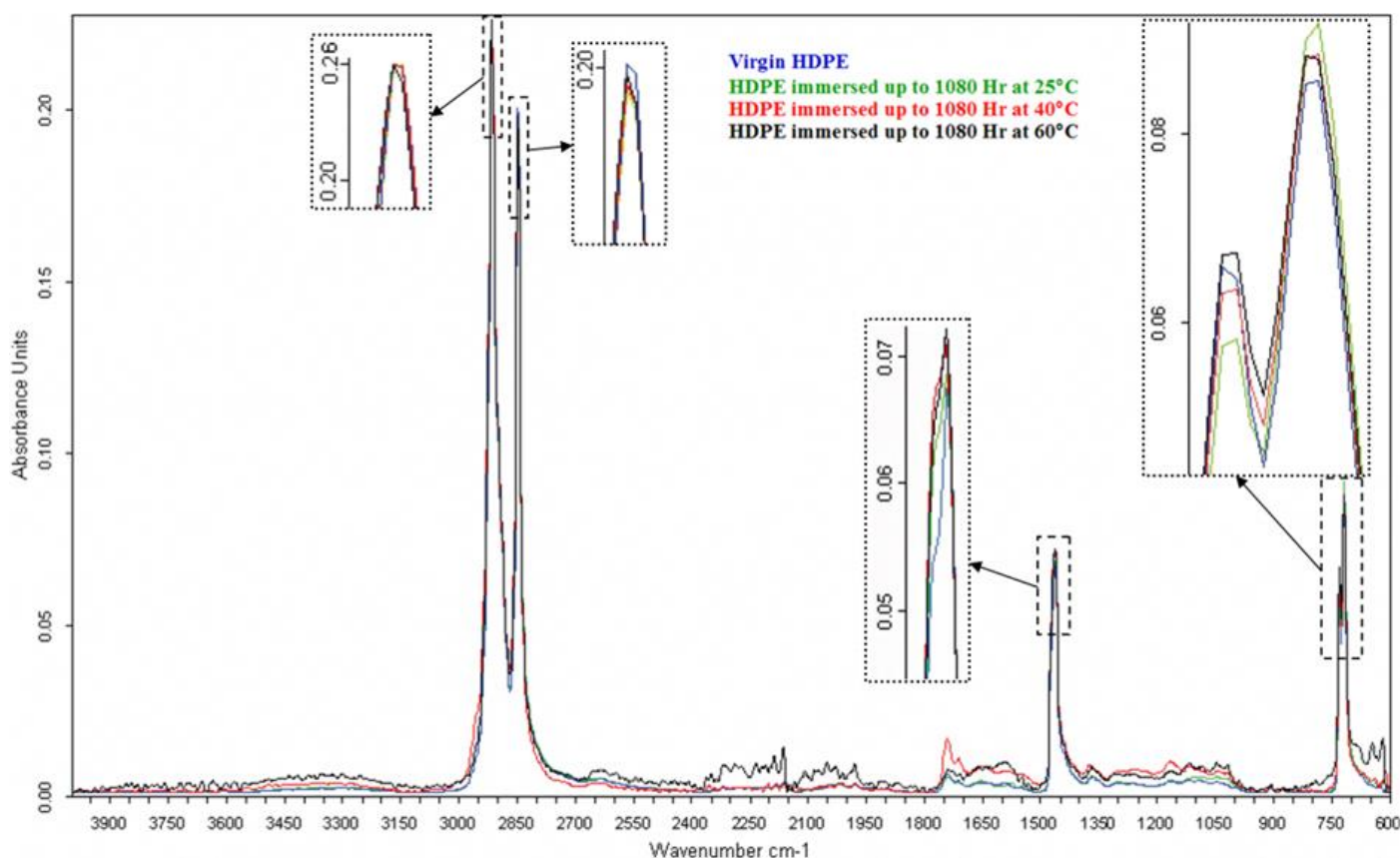
The main bands of the high-density polyethylene under investigation include: a band located at approximately 2900 cm^{-1} can be attributed to CH_2 as asymmetric stretching; a bending deformation can be seen in a band at approximately 1462–1472 cm^{-1} ; and there's also a band at 719–730 cm^{-1} that exhibits a rocking deformation [3, 6]. In the spectra (at 40 & 60 °C), an asymmetric band with a core at 1600 cm^{-1} could also be seen. This is consistent with the bending modes of the absorbed water. Likewise, water molecules are responsible for the OH^- stretching band at 3400 cm^{-1} . Guermazi et al. [3, 6] was also showing the same results previously.

The only significant difference between the spectra corresponding to the unaged and aged samples is the intensity of the bands: The signal intensity is weaker and there are fewer specific groupings in the aged specimen.

For example, the intensity of all subsequent absorptions (equivalent to 2848 cm^{-1} , 730 cm^{-1} , etc.) was reduced. This marks the beginning of the disappearance of these bands.

Table 1 Examined FTIR spectral bands

Wave number (cm ⁻¹)	Functional groups	Type of vibration	Ref.
719 and 730	CH ₂	Rocking deformation	[28-30]
1117	C=O	C=O stretching	[20]
1462 and 1472	-CH ₂ -	Bending deformation	[20]
1715	(RR') > CO	C=O stretching where R and R' are aliphatic groups	[19,31]
1738	R-CO-OR'	C=O stretching where R and R' are alkyl groups	
2848	CH ₂	CH ₂ symmetric stretching	
2916	CH ₂	CH ₂ asymmetric stretching	[3,19]
3400	O-H	Water molecules	[3,31]

**Figure 6.** HDPE spectra acquired at absorption mode for unaged and aged test pieces at 25, 40, 60 °C over 1080 Hr.**Table 2** Distinctive spectral bands as a function of aging temperature corresponding to generated, transformed or unchanged groups

HDPE immersed up to 1080 Hr at	Intensity increase generated groups (cm ⁻¹)	Intensity decrease transformed groups (cm ⁻¹)	Unchanged intensity
25°C	719, 1117, 1472, 2916	730, 1600, 1715, 1738, 2848	1161, 1368, 1462, 3400
40°C	719, 965, 1117, 1161, 1368, 1462-1472, 1600, 1715, 1738, 2916, 3400	730, 2848	
60°C	719, 730, 1117, 1161, 1368, 1462-1472, 1600, 1750, 1738, 2916, 3400	2848	

Table 3 Chain breaking and chain branching-related spectral band change for three aging temperatures

Inv: invariant; +: increase; -: decrease; w: weak increase

HDPE immersed up to 1080 Hr at	719	730	1049	1117	1161	1368	1462	1472	1600	1715	1738	2848	2916	3405
25°C	++	--	+	+	Inv	Inv	+	+	-	w	-	-	w	+
40°C	+	-	+	++	+	+	+	+	+	++	+	-	w	+
60°C	+	+	+	++	+	+	+	+	++	+	++++	-	Inv	+

In both cases, the intensity change is sufficient to reveal the initial indications of impending cleavage and breaking of the polymer chain. Such structural changes can undoubtedly be related to an effect of aging. This result again confirms the previously documented mechanical degradation as well as the negative effects of immersion aging at different temperatures.

3.3. SEM examination of HDPE specimens

To confirm the previously noted mechanical degradation, we examined microscopic observations of the aged materials using scanning electron microscopy (SEM) typically used for observation of specimen surfaces. Figure.7 illustrates the fracture surface morphology of unaged HDPE studied at four different scales (2 mm, 0.5 mm, 0.2 mm, 10 μ m).

From observation of Figure.7, it can be seen that the fracture surface of the unaged HDPE sample has a relatively normal appearance. Referring to Figure.7. b, which is a 500 μ m enlargement of area B (shown in Figure .7. a), it can be concluded that the sample fibers show no signs of fracture phenomena. Similarly, by examining Figure.7. c, which represents a 200 μ m enlargement of zone C (described in Figure.7. b), the structural fibers in this zone show no evidence of rupture. Figure.7. d illustrates a zoom by 10 μ m of the area D. It is therefore clear from analysis of this figure that the unaged

samples show no damage at the microscopic level of the material.

A formation of huge cavities in the matrix of the samples exposed to boiling water was noted by Birger et al. [13] in their study about the effects of hygrothermal aging on the fracture processes of graphite-fabric epoxy composites. The same morphological changes could be reported by Zhao et al. [22] for EPDM samples (ethylene propylene diene monomer) under artificial weather conditions created by fluorescent UV. Also, Aglan et al. [23] observed ridges and surface irregularities in fracture surface morphologies of hygrothermally aged polyurethanes. Yang et al. [24] studied the morphological changes of polyurethane coatings exposed to water. They have discovered that microscale filamentous threads grew in the coated surface during immersion, making it appear rougher due to surface degradation. Similarly, Zhou and Lucas [25] have found cracks and cavities in graphite/epoxy composites immersed in water at 75 and 90 °C for 4300 hours.

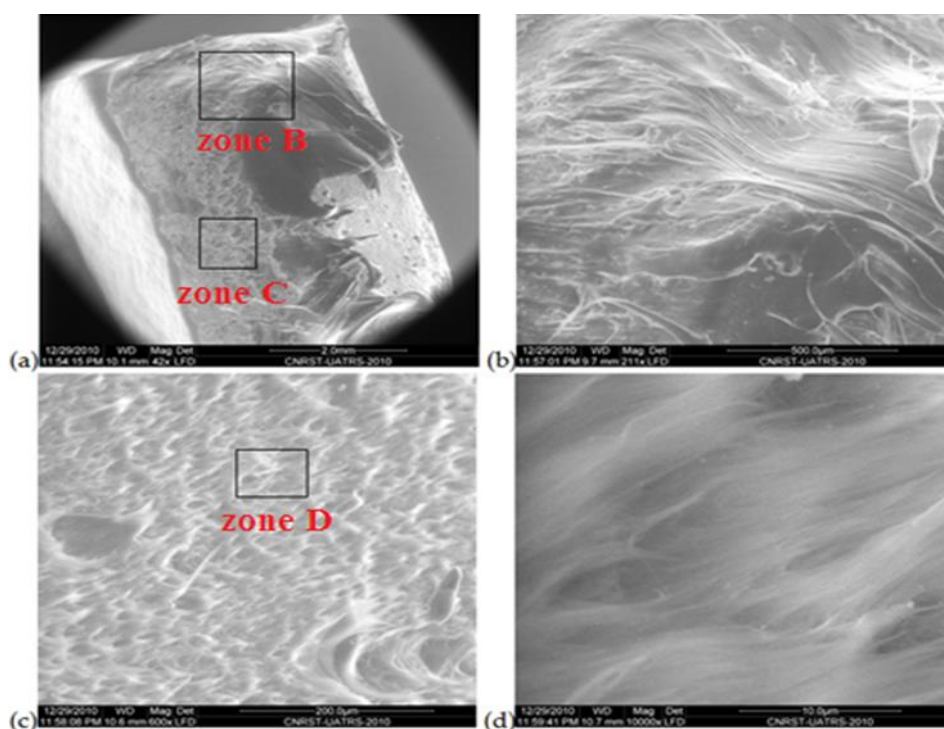


Figure 7. Fracture surface photos of unaged HDPE. SEM images a (a) 2 mm, (b) 0.5 mm [zone B], (c) 0.2 mm [zone C], (d) 10 μ m zoom for [zone D] scale

Figure.8 depicts the fracture surface morphologies of HDPE subjected to 1080 hours of hydrothermal aging at a temperature of 25°C. Examination of Figure.7. b, which shows a 500 µm enlargement of zone B (shown in Figure 7.a), reveals the state of damage after a tensile test of HDPE that has undergone chemical attack, at the level of the outer volume of the material (lateral and subjacent surface of the sample). This analysis suggests that the first signs of hygrothermal ageing at 25°C are

beginning to appear, in comparison with the unaged samples shown in Figure.7.

According to the observations in Figure.9. b, which offers a magnification of 0.2 mm on zone B (shown in Figure.9. a), as well as Figure.9. c, exhibiting a magnification of 0.4 mm on zone C (shown in Figure.9. a), and finally Figure.9. d, showing a magnification of 0.5 mm on zone D (also from Figure.9. a), it is possible to discern the appearance of

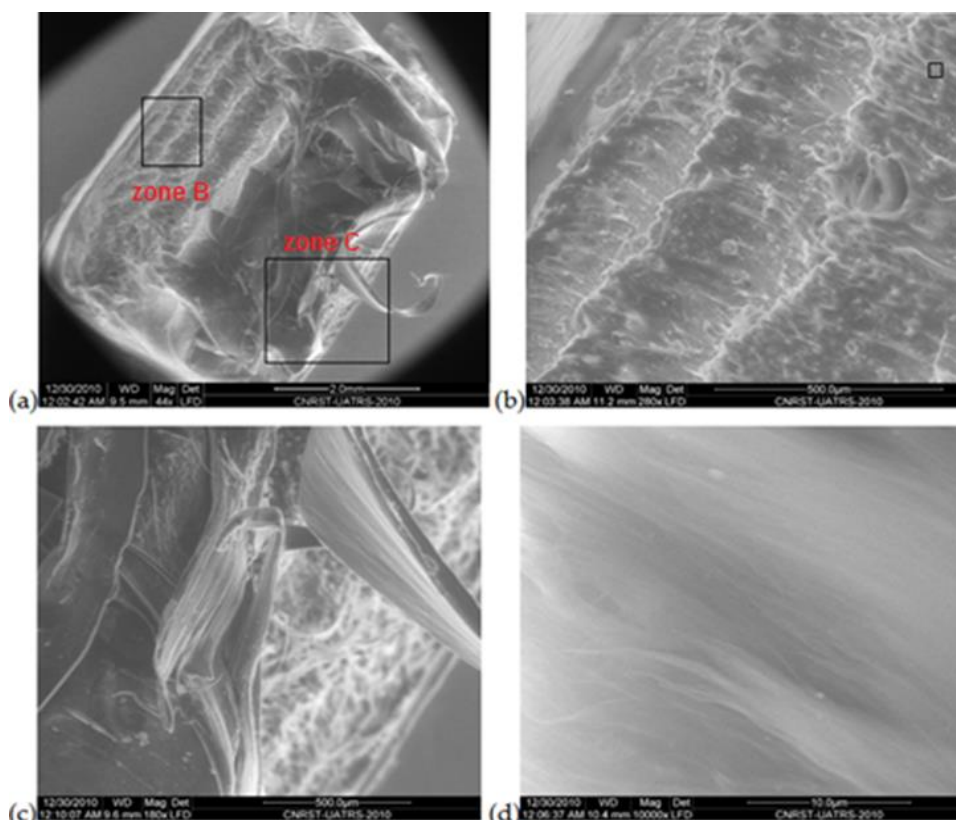


Figure 8. Fracture surface photos of HDPE aged at 25 °C for 1080 Hr. SEM images a (a) 2 mm, (b) 0.5 mm [zone B], (c) 0.5 mm [zone C], (d) 10 µm scale.

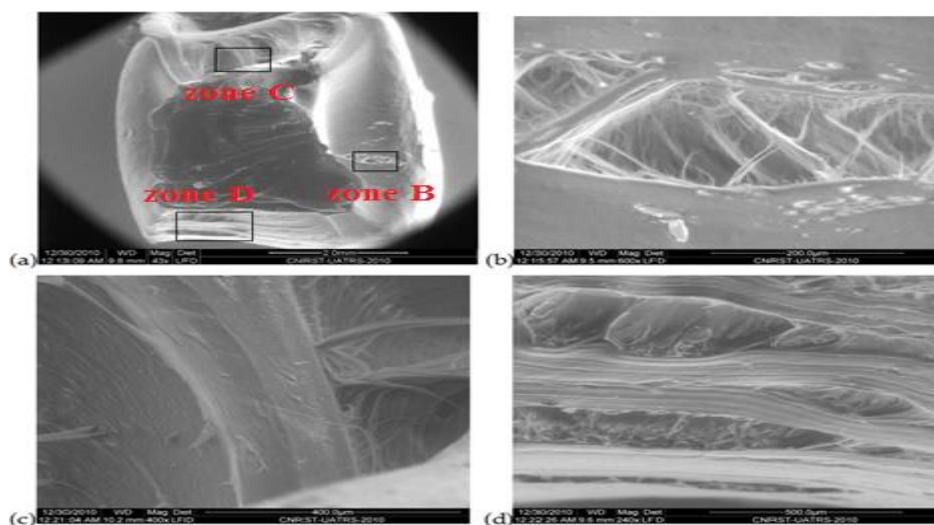


Figure 9. Fracture surface photographs of HDPE aged at 40°C for 1080 Hr. SEM images a (a) 2mm, (b) 0.2 mm [zone B], (c) 0.4 mm [zone C], (d) 0.5 mm [zone D] scale.

cracks emerging in a direction perpendicular to the aligned fibrils under the effect of tensile stress.

In the light of these findings, it is clear from Figure.9 that the first signs of the aging effect at 40°C are becoming apparent. The sample undergoes sudden failure due to loss of elasticity.

Figure.10 shows an electron micrograph of the cross-section of an HDPE sample aged at 60°C and broken during a tensile test. Referring to Figure.10. b, which

details zone B at 0.2 mm magnification (Figure.10. a), and to Figure.10. c and Figure.10. d, which respectively represent a 0.2 mm and 10 μm magnification of zone C (also from Figure.10. a), it is possible to distinctly observe cracks developing perpendicular to the direction of tensile stress, covering approximately 450 μm . Consequently, it is clear from these observations that when the aging temperature reached 60°C, cracks formed, affecting the surface of the upper neck area. In addition, the loss of elasticity was found to increase in correlation with the rise in exposure temperature.

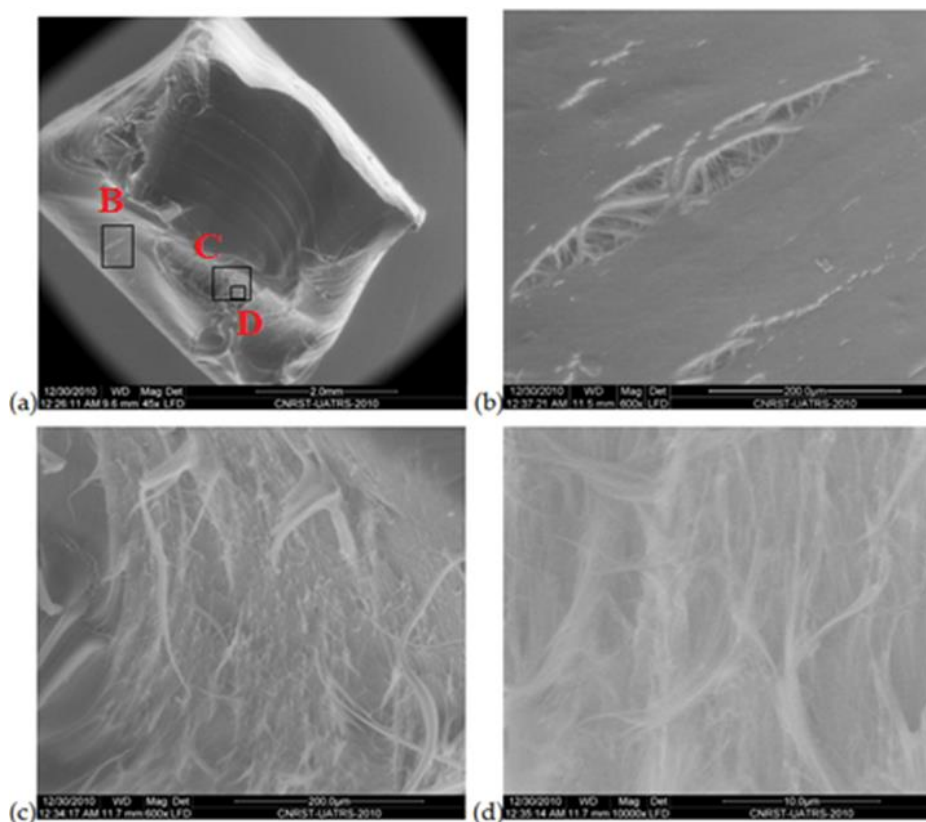


Figure 10. Fracture surface photographs of HDPE aged at 60°C for 1080 Hr. SEM images a (a) 2mm, (b) 0.2 mm [zone B], (c) 0.2 mm [zone C], (d) 10 μm [zone D] scale.

The influence of aging in sulfuric acid at different temperatures on the failure mechanisms of HDPE was investigated. The phenomenon of sorption has been studied [26], and solvents act mainly through diffusion of solvent molecules within polymer systems, causing plasticization of HDPE. The results of the previously obtained tensile test [26, 6, 27] showed that the stress and elongation at break decreased depending on the spreading of the solvent in the HDPE sample and reached a minimum when the saturation with the solvent in the sample was reached.

Loss of elasticity and diffusion kinetics are closely correlated. The loss of elasticity, which reaches 42 %, is reversible. When the temperature and the duration of immersion are high, this phenomena is even more significant. This demonstrates the sulfuric acid

solution's (pH=1) extreme aggression and ability to more readily and abundantly penetrate between macromolecules. It destroys secondary bonds between them and increases molecular mobility. The thermal penetration is what causes the polymer to become brittle and can cause bleeding of the organic compounds or even cracks or fissures.

All of these mechanisms are controlled by the cohesive energy and significantly modify the mechanical properties.

4. CONCLUSION

In this work, the impact of aging factors on the mechanical properties of the HDPE material was examined. To model aging processes, various immersion periods and aging temperatures were used. The results confirm that chemical

and structural modifications are taking place in the High-Density Polyethylene polymer chains.

In terms of mechanical characteristics, it should be underlined that the specimen inflexibility increases with higher immersion time and aging temperature. This means a reduction in the breaking stress and above all a reduction in the breaking elongation. This supports the theory that the development of carbonyl groups in the polymer's structural core represents the significant chemical shift caused by the loss of elasticity and ductility.

The microscopic observations of the SEM analysis confirmed the loss of elasticity. The degradation of the immersed material appears mainly in form of cracks after a short period of immersion and the deterioration of the material under severe conditions. Finally, the aging temperatures combined with the sulfuric acid solution appear to be the main causes of HDPE deterioration.

REFERENCES

- [1] Greig, JM., *Plast Rubber Compos.* vol 21, (1994) pp.133–40.
- [2] Han, MH., Nairn, JA. *Compos. Part A: Appl. Sci. Manuf.* vol 34, issue 10 (2003) pp.979–986.
- [3] Guermazi, N., Elleuch, K., Ayedi, HF. *Materials and Design.* vol 30, issue 6 (2006) pp.2006–2010.
- [4] Salem, A., Bensalah, B., Mezlini, S. *Polym testing.* Vol 94, (2021)
- [5] Liu Lulu, Zhao Zhenhua, Chen Wei, Shuang Chao, Luo Gang. *Composites structures.* Vol 204, (2018) pp.645–657
- [6] Guermazi, N., Elleuch, K., Ayedi, HF., Kapsa PH. *Journal of Materials Processing Technology.* vol 203, issue 1-3 (2008) pp.404–410.
- [7] El brahmi A, Abderafi S. *Materials today: Proceeding.* vol 27, (2020) pp.3028–3032
- [8] Bo wu, Feifei liu, Wenwen Fang, Tony yang, Guang-Hao Chen, Zhili He, Shanquan Wang. *Science of the total environment.* Vol 778, (2021) pp.146–85
- [9] Sahar Foorginezhad, Masoud Mohseni-Dargah, Khadijeh Firoozirad, Vahid Aryai, Amir Razmjou, Rouzbeh Abbassi, Vikram Garaniya, Amin Beheshti, Mohsen Asadnia. *Process Safety and Environmental Protection.* Vol 147, (2021) pp. 192–213
- [10] Nielsen, A.H., Vollertsen, J., Jensen, H.S., Wium-Andersen, T., Hvitved-Jacobsen, T. *Water Research.* Vol 42, (2008) pp.4206–4214
- [11] Lasfar, S., Mouallif, I., Latrach, A., Choukir, A., Diab, A. *Journal of Materials and Environmental Science.* vol 6, issue 11 (2015) pp.3002–3014.
- [12] Mouallif, I., Latrach, A., Chergui, M., Hangouët, J.-P., Barbe, N. *Journal of Composite Materials.* vol 48, issue 24 (2014) pp.3025–3034.
- [13] Birger, S., Moshonov, A., Kenig, S. *Composites.* vol 20, issue 4 (1989) pp.341–8.
- [14] Zanni-Deffarges, MP., Shanahan, MER. *International Journal of Adhesion and Adhesives.* vol 15, issue 3 (1995) pp.137–42.
- [15] Christenson, EM., Erson, JM., Hiltner, A., Baer, E. *Polymer.* vol 46, issue 25 (2005) pp.11744–11754
- [16] Ceretti, ED., Felder, E., Haudin, JM. *Annales de Chimie Science des Matériaux.* vol 28, issue 3 (2003) pp.1–14.
- [17] Chandra, R., Rustgi, R. *Polymer Degradation and Stability.* vol 56, issue 2 (1997) pp.185–202.
- [18] Pegoretti, A., Migliaresi, C. *Polym Compos.* vol 23, issue 3 (2002) pp. 342–51.
- [19] Wagner, HD., Lustinger, A. *Composites.* vol 25, issue 7 (1994) pp.613–6.
- [20] Rui Yang, Ying Liu, Jian Yu, Kunhua Wang. *Polymer Degradation and Stability.* vol 91, issue 8 (2006) pp.1651–1657.
- [21] Chen, Y., Davalos, JF., Ray, I., Kim, HY. *Composite Structures.* vol 78, issue 1 (2007) pp.101–11.
- [22] Zhao, Q., Li, X., Gao, J. *Polymer Degradation and Stability.* vol 92, issue 10 (2007) pp.1841–6
- [23] Aglan, H., Calhoun, M., Allie, L. *Journal of Applied Polymer Science.* vol 108, issue 1 (2008) pp.558–64.
- [24] Yang, XF., Tallman, DE., Croll, SG., Bierwagen, GP. *Polymer Degradation and Stability.* vol 77, issue 3 (2002) pp.391–6.
- [25] Zhou, J., Lucas, JP. *Composites Science and Technology.* vol 53, issue 1 (1995) pp.57–64.
- [26] Mouallif, I., Latrach, A., Chergui, M., Benali, A., Barbe, N. 20ème Congrès Français de mécanique, CFM (2011), 824.
- [27] Domenech, SC., Severgnini, VLS., Pinheiro, EA., Avila, AOV., Borges, NG., Lima, E., Drago, V., Soldi, V. *Polymer Degradation and Stability.* vol 88, issue 3 (2005) pp.461–467.
- [28] Ilaria Donelli, Giuliano Freddi, Vincent A. Nierstrasz, Paola Taddei. *Polymer Degradation and Stability.* vol 95, issue 9 (2010) pp.1542–1550.
- [29] Gaur, U., Chou, CT., Miller, B. *Composites.* vol 25, issue 7 (1994) pp.609–12.
- [30] Rui Yang, Ying Liu, Jian Yu, Kunhua Wang. *Polymer Degradation and Stability.* vol 91, issue 8 (2006) pp.1651–1657.
- [31] Zanni-Deffarges, MP., Shanahan, MER. *International Journal of Adhesion and Adhesives.* vol 15, issue 3 (1995) pp.137–42.
- [32] Prakash Bhuyar, Nurul Aqilah Binti Mohd Tamizi, Mohd Hasbi Ab. Rahim, Gaanty Pragas Maniam, Natanamurugaraj Govindan. *Maejo International Journal of Energy and Environmental Communication.* Vol1, issue 2 (2019) pp.26–31.