

Resistance of different materials used in sewers systems: Polyvinyl chloride (PVC), polypropylene (PP) and High density polyethylene (HDPE), to sulfuric acid and sodium sulfate attack.

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ABSTRACT

The behaviour of PVC, PP and HDPE used in sewer systems exposed to acid and sulfate solutions was investigated at 25°C and 40°C. Gravimetric characterization proves that PVC has a fickian behavior. It shows also, that PP has a non-fickian behavior, characterized by a rapid acceleration of water absorption, and the HDPE has a fickian behavior at 25°C, while it has a non-fickian behavior at 40°C, characterized by a weight loss after a certain aging period. The prolongation of the time of exposure to sulfuric acid solution leads to a progressive increase in tensile strength followed by a slight decrease at 40°C. The unaged samples of all materials have a much lower tensile strength than those of the aged samples, and a drop in elongation at break could be observed. These results can be explained by the increase of crystallinity, followed by the increase of crosslinking density due to the diffusion of the solvent and the effect of the temperature as the exposure time increases. These results prove that there is a correlation between the diffusion of the solvent as well as the increased exposure time and temperature on the mechanical properties of polymer.

Keywords - Mechanical properties, HDPE, PP, PVC, sodium sulfate, sulfuric acid, water absorption.

I. Introduction

Polyvinyl chloride (PVC), polypropylene (PP) and High density polyethylene (HDPE) are the polymeric materials used in the wastewater pipes. This polymer material becomes more and more dominant in the pipe industry. It provides a high performance, a durable and economic solution. The durability of polymer materials has a primary importance [1-7].

The degradation of polymers is provoked by the chemical attack, as it is well known; the deterioration of materials used in sewers systems is a major problem. As reported by [1-7,8-10] hydrogen sulfide produced by anaerobic wastewater is released into the wastewater flow, where it spreads to the atmosphere and condensates on the pipe wall above the wastewater. Micro-organisms, such as Thiobacillus thiooxidans, growing on this surface aerobic (presence of oxygen) convert the hydrogen sulfide into sulfuric acid. Corrosion

deterioration has long-term effects on the environment, the economy and the society [8].

Other various environment factors lead to the deterioration of the properties of polymeric materials, e.g., humidity, elevated temperature, UV radiation, oxidative degradation, mechanical stress, etc. Regardless of the origin of the degradation, it always affects their physical and mechanical properties causing the reduction of their durability [1-7,11,12,14].

Before using polymer, we must have an idea about their chemical, as well as physical and morphological behavior in their environment, because these characteristics of polymer can have a great influence on their mechanical properties. However, their exact effects on the hydrothermal behavior of this type of materials have not been widely discussed in the literature [1-7].

MOUALLIF et al. [1,2] have investigated the effect of thermal ageing in sulfuric acid solution of HDPE

pipe. They have found that the diffusion of solvent into HDPE sample and the mechanical properties largely depends on the aging temperature.

Weon et al. [14] have investigated the effects of thermal ageing on mechanical of LLDPE pipe. They have found that long-term exposure to an elevated temperature causes a deleterious effect on mechanical properties by the increase of crystallinity and crosslinking density.

Guermazi et al. [15] have studied the effect of time and aging temperature on structural and mechanical properties of pipeline coating using synthetic sea water. They have found that the mechanical properties are more affected as the aging temperature increases, also they have shown that there is a perfectly relation between the evolution of the mechanical properties, structural modifications and chemical changes.

This article aims to acquire more information on the toxicity of wastewater and its impact on the studied material. To achieve this goal, the laboratory-accelerated tests involved immersion of PVC, PP and HDPE in sulfatic solutions (H_2SO_4 , Na_2SO_4) at two different temperatures (25, 40°C), to study how the thermal ageing and time exposure using these solutions has an effect on the mechanical properties.

II. Experimental

2.1 Tested Materials

The materials tested in this study are: PVC, PP and HDPE, normally used in sewage systems. The VICAT softening temperature (VICAT) of PVC was carried out using LLOYD Instruments material testing machine, as per NM EN 727 2013. The Melt

Index (MI) can be defined as the weight of the polymer (in grams) extruded in 10 min through a standard orifice at prescribed temperature and pressure [13,14]. The MI measured of HDPE and PP was carried out using a LLOYD Instruments material as per ISO 1133. HDPE and PP melt was extruded through the orifice at a load of 5 kg at 190°C. The density of PVC, PP and HDPE was measured (0.0001 g) using a SARTORIUS LA310S balance per NM ISO 1183-1. The measurements of VICAT, Melt Index (MI) and density are given in "Table 1".

Samples from PVC sewer pipe ($\varnothing 400 \times 300 \times 10 \text{ mm}^3$) were removed from the longitudinal direction. Samples from PP and HDPE annulated sewer pipes ($\varnothing 1000 \text{ mm}$) were removed from the transversal direction of the inner wall. All standard specimens were obtained as per NM ISO 6259-2.

2.2 Exposure

The chemical resistance of the polymer samples to the following types of attack was investigated at room temperature $25 \pm 2^\circ\text{C}$ and at $40 \pm 1^\circ\text{C}$:

- Continuous immersion in 4 liter of sulfuric acid (H_2SO_4) solution 5000ppm of SO_4^{2-} , with a pH= 1. The maximum immersion time considered in this test is equal to 307 days at 25°C .
- Continuous immersion in 4 liter of sodium sulfate (Na_2SO_4) solution 5000ppm of SO_4^{2-} , with pH= 7. The maximum immersion time considered in this test is equal to 307 days at 25°C .

Table 1: Measurements of VICAT, Melt Index and density for our materials

Materials	PVC	PP	PEHD	Standard
Density at 23°C (g/cm^3)	1,439	0,9139	0,9619	NM ISO 1183-1
Indice de fluidité ($\text{g}/10\text{min}$)	—	MFR(190°C , 5Kg) = 0,7131	MFR(190°C , 5Kg) = 2,3788	ISO 1133
VICAT ($^\circ\text{C}$)	85°C .	—	—	NM EN 727 2013

2.3 Gravimetric measurements

Every week, the weight of each sample was measured. After drying the samples with a cloth, their weight was recorded using a technical balance with a sensitivity of 0.0001 g.

The weight gain $m(t)$ of each specimen was calculated from its initial weight $M(0)$ and its weight after absorption $M(t)$ as follows "equation 1" [1-7,11-13]:

$$m(t) = \frac{M(t) - M(0)}{M(0)} \times 100 \quad (1)$$

2.4 Tensile test

The effect of ageing in sulfuric acid effect on the mechanical properties of the different polymer materials was characterized by tensile testing performed at room temperature.

The tensile tests followed the guidelines of EN ISO 6259-1 standard. The tensile test was carried out using a Zwick Roell Z010 testing machine. An extensometer was employed to measure the displacement in the gauge length region.

The PVC specimens were stretched at the strain rate of 5 mm/min till rupture. The PP and HDPE specimens were stretched at the strain rate of 100 mm/min till rupture.

III. Results and discussion

3.1 Gravimetric measurements

"Fig.1" shows the evolution of the mass of the specimens of PVC, PP and HDPE immersed in the sulfuric acid (H_2SO_4) depending on the square root of time at 25°C and 40°C .

For illustration, we have separated the results into distinct graphs. One shows the results at 25 °C and the other at 40°C.

According to the absorption curves of PVC aged in H₂SO₄ and in Na₂SO₄ at 25°C and 40°C "Figs. 1. a), b), c) and d)", we found that PVC shows a fickian behavior, characterized by:

- A first part corresponding to a rising phase of water absorption, where the weight of the sample is increasing, which indicates a penetration of water into the amorphous parts of the material to saturation [1,2,17]
- A second part is relatively stable, that characterizes a 'pseudo plate' of diffusion.

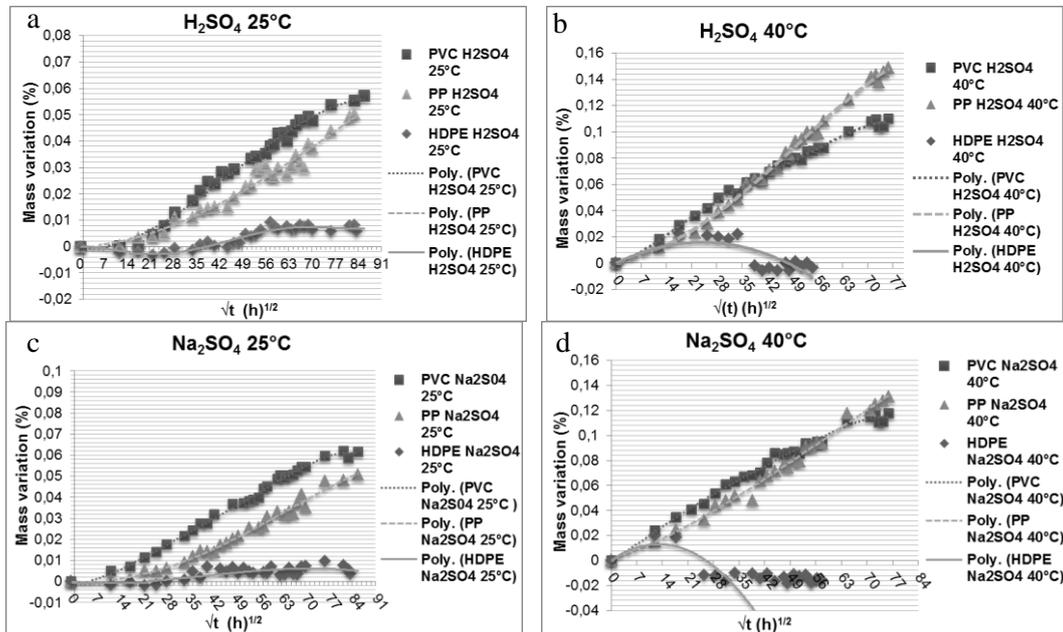


Fig. 1 Weight change as a function of time (\sqrt{t} (h^{1/2})) for PVC, PP and HDPE exposed to: sulfuric acid solution (H₂SO₄) a) at 25°C, b) at 40°C and sodium sulfat c) at 25°C, d) 40°C

Furthermore, according to the absorption curves of PP aged in H₂SO₄ and in Na₂SO₄ at 25°C and 40°C "Figs. 1. a), b), c) and d)", we found that PP has a non-fickian behavior, characterized by a rapid acceleration of water absorption, which is usually accompanied by a large deformation and damage within the material [1,2,18]. Moreover, we found that the absorption increases with increasing temperature.

Finally, according to the absorption curves of HDPE aged in H₂SO₄ and in Na₂SO₄ at 25°C and 40°C "Figs. 1. a), b), c) and d)", we discovered that HDPE at room temperature has a fickian behavior, while it has a non-fickian behavior at 40 °C. In each figure, we can observe a linear part and a part of saturation, while a third part is corresponding to a descending phase, where the weight of the sample decreases gradually and finally becomes negative. These behaviors can be ascribed to many phenomena such as, the internal reorganization of polymer chains, the leaching of certain low molecular weight components of the material, and the migration of cross-linking agents to the surface during aging process [1,2,12]. Similar results have been reported in the literature [1,2,12].

3.2 Determination of the diffusion coefficient

Although it is not a completely fickian absorption across the aging, we assume that the first part of the

curve is the linear function of the square root of time satisfies these laws in the literature [1, 2,16].

As for the case of PP aged at 25°C and 40°C in both solutions, we discovered that the material does not reach the level of absorption in the time span examined. The weight gain of the samples was regularly throughout the growing aging at 25 and 40°C, and more pronounced at 40°C. The determination of $m(\infty)$ in this case is difficult. However, on aging time ($t^{1/2} < 70$ h^{1/2} at 25°C and $t^{1/2} < 60$ h^{1/2} at 40°C), we came to a first approximation, using Fick's laws to describe this phenomena.

In the case of HDPE aged at 40°C in H₂SO₄ and Na₂SO₄, the mass $m(\infty)$ is determined before the mass decrease. This means that most of the absorption is completed before the phenomenon of decline [1,2,17]. Thus, from the slope of mass variation $m(t)$ versus the square root of time at each temperature and at each test solution, D can be evaluated by using the "equation 2" [1,2,16,17,19]:

$$\frac{m(t)}{m(\infty)} = \frac{M(t) - M(0)}{M(\infty) - M(0)} = \frac{4}{h} \sqrt{\frac{Dt}{\pi}} \quad (2)$$

Where:

- $m(t)$ = mass of water (or the solute) absorbed at time t
- $m(\infty)$ = estimation of the mass (or the solute) absorbed at saturation (assuming a fickian behavior).

And: $M(0)$ is the initial weight of sample, $M(\infty)$ is the estimation of the weight of the sample at saturation (assuming a fickian behavior), and h is the samples thickness.

"Table 2" summarizes the diffusion coefficients, the estimated mass of saturation and the saturation time of the studied materials PVC, PP and HDPE aged in H_2SO_4 and Na_2SO_4 at $25^\circ C$ and $40^\circ C$, obtained by exploiting the values of $m(\infty)$ estimated.

Table 2: Diffusion coefficient D ($\times 10^{-6} \text{ mm}^2/\text{s}$), estimation of the mass at saturation $m(\infty)$ (%), and saturation time t_s (h) observed for the materials HDPE, PP and PVC immersed in distilled water + sulfuric acid and distilled water + sodium sulfate at different temperatures.

Materials	Temperature T = 25°C						Temperature T = 40°C					
	H_2SO_4			Na_2SO_4			H_2SO_4			Na_2SO_4		
	D	$m(\infty)$	t_s	D	$m(\infty)$	t_s	D	$m(\infty)$	t_s	D	$m(\infty)$	t_s
HDPE	0,20	0,008	3432	0,20	0,007	3576	1,5	0,02	312	3,01	0,02	144
PP	0,34	0,03	3576	0,32	0,03	3816	0,46	0,1	2832	0,47	0,09	2832
PVC	0,84	0,054	5760	0,88	0,06	5760	1,07	0,11	5040	1,07	0,115	5040

According to "Table 2", we found that for both types of aging solution (H_2SO_4 and Na_2SO_4), each material at each temperature has almost the same mass value of saturation. At the level of saturation time, we found that the saturation time decreased with increasing temperature. In the same manner we could discover, that we obtained values for the diffusion coefficients of our materials, which are very close, as well as an increase in these coefficients with increasing in temperature. An exception is the case of HDPE at $40^\circ C$, for which a difference between the values of the diffusion coefficients, as well as a an elevated value in the Na_2SO_4 solution was noted, an effect which is unexpected for this material, that is characterized by a low absorption compared to the other two materials in room temperature in both solutions. We can explain this difference, by the effect of temperature which accelerates the absorption followed by the mass decrease, due to a reorientation and internal leaching of a component of the material as already defined.

3.3 Arrhenius treatment

The phenomena of water diffusion in polymers as a thermo-active process, the diffusion rate is a function of temperature that obeys a type of Arrhenius law "equation 3": [1,2,16-18]

$$D = D_0 \exp \frac{-E_a}{RT} \quad (3)$$

This equation was used to estimate the activation energy for the water diffusion process.

Where E_a , D_0 , R and T are respectively the activation energy for diffusion, a constant coefficient, the universal gas constant ($R=8, 32 \text{ E}^{-3} \text{ KJ.mol}^{-1}$), and the temperature in degrees Kelvin. This equation can be reduced to a linear form by taking the natural logarithm of both sides [16].

From the slope ($-E_a/R$) of the resulting curves, the activation energy (E_a) can be determined. The results

obtained after immersion in both H_2SO_4 and Na_2SO_4 are shown in "Fig. 2".

Therefore, a graph of $\ln(D)$ versus $1/T$ should give us a negative line slope and $-R/E_a$ value.

For clear illustration, we have separated the results into distinct graphs. One shows the results for PVC and PP and the other for HDPE.

Normally, the calculation of activation energies need at least three different test temperatures [1-7,12], but in our stuy we calculated these energies only just to compare the difference between these energies for each environment.

"Fig. 2" shows the variation of $\log D$ versus $1/T$ for the PVC, PP and HDPE.

The activation energy, calculated by the slope of $\log D$ versus $1/T$ "Fig. 2", provided us an activation energy of about 12.6 KJ/mol in the case of immersion in H_2SO_4 and an energy E_a of about 10.3 kJ/mol in the case of immersion in Na_2SO_4 . For the PP specimen we observed an activation energy of about 22.1 kJ/mol for the test specimens aged in H_2SO_4 while the energy is about 20 KJ/mol in the case of specimens aged in Na_2SO_4 . For HDPE we found an energy of activation of about 103 KJ/mol in the case of immersion in H_2SO_4 and about 140 KJ/mol in the case of immersion in Na_2SO_4 . We note, that the tests conducted with specimens of PVC and PP show close values of activation energy for both solutions, with an increasing of 2KJ/mol for the energy required for the diffusion of the acid solution. Our activation energy for the PP specimen is in perfect agreement with the results found in the scientific literature, such as [16] where the activation energy of reinforced polypropylene-clay nanocomposites in distilled water is between 25.3 KJ /mol and 35.5 KJ/mol with an increase of 4 kJ /mol in the case of immersion in a solution of seawater.

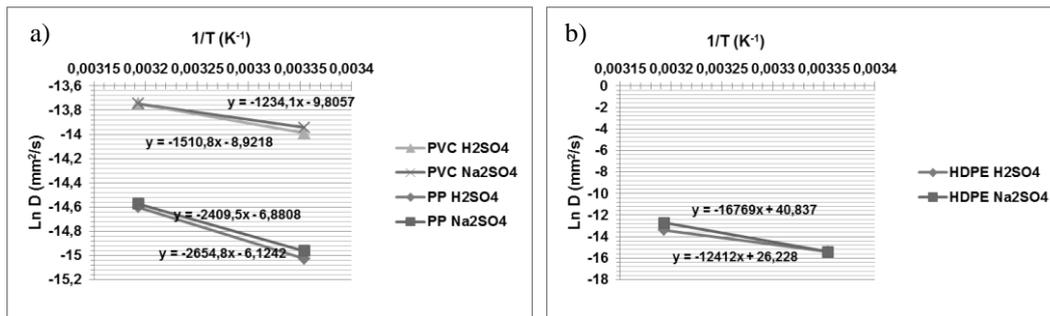


Fig. 2 Estimation of the activation energy of the diffusion process in H₂SO₄ and in Na₂SO₄: a) for PVC and PP, b) for HDPE

Furthermore, for HDPE, the activation energy required for the diffusion of sulfate is higher than the energy for the acid diffusion. This result can be explained, as already mentioned, by the effect of the temperature which accelerated the absorption followed by a rapid decrease in mass. The activation energies, which were estimated for the aging in acid and sulfate solution are too elevated. Normally the activation energy of the diffusion process of water in polyethylene is about 30 kJ/mol [17,20] or even of the order of 39.1KJ/mol [1,2] when immersed in sulfuric acid.

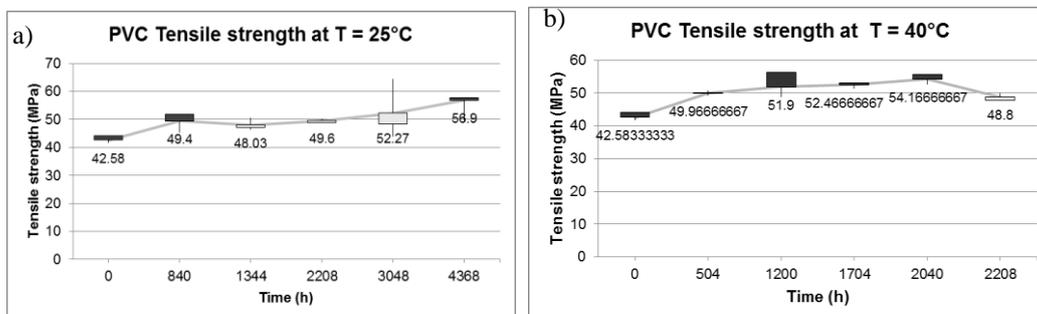
The divergence of results is due to the high diffusion coefficient obtained in the case of immersion in the sulphate at 40°C and to the fact that the most authors worked with three different test temperatures, while we found that the diffusion coefficients in the acid solution is close. For example we obtained a diffusion coefficient in acid equal 0.20×10^{-6} mm²/s at 25°C and 1.5×10^{-6} mm²/s at 40°C, these results are too close to the coefficients calculated by [1,2] which they found 0.88×10^{-6} mm²/s at 25°C and 1.86×10^{-6} mm²/s at 40°C, as well as an additional value measured at 60°C, which allows to find $E_a = 39.1$ KJ/mol.

3.4 Mechanical behavior

The mechanical properties of specimens of PVC, PP and HDPE immersed at sulfuric acid solution (H₂SO₄) at room temperature and at 40°C during a certain period of time were investigated. The key mechanical

properties (i.e., tensile strength and elongation at break) are shown in "Figs. 3, 4 and 5" for PVC, PP and HDPE successively.

According to "Figs. 3, 4 and 5", we found that the non-aged samples of PVC, PP and HDPE have a lower tensile strength than the specimens aged in H₂SO₄. As in the case of material aged at 25°C, we found an increase in tensile strength as a function of exposure time for all of the three materials, while we found for the case of PVC aged at 40°C, that the resistance increased up to 2040 h. Thereafter, a slight decrease in resistance could be observed. In the same way, an increase in resistance until 1704 h, followed by a decrease in strength for the PP samples, and an increase in resistance up to 2208h with following decrease for the HDPE samples could be noted. Even though the resistance of these three materials decreases, it remained higher than the initial value during the test. However, there is also a slight regression of elongation at break; each material with a similar variance. So we observed for a period of aging equal to 4000h or more in the acid at 25°C a drop of 74.81% to 126.2% for PVC, a drop of 706.53% to 308.83% for the PP and a drop of 386.43% to 100.99% for HDPE. On the other hand, we found for an aging time of less than 4000h in the acid at 40°C a drop 126 126.2% to 18.1% for PVC, a drop of 706.53% to 61.3% for PP and a drop of 386.43% to 100.6% for HDPE. We conclude that the materials aged in sulfuric undergo a sharp drop in the elongation function of the aging time and temperature depending on the acid.



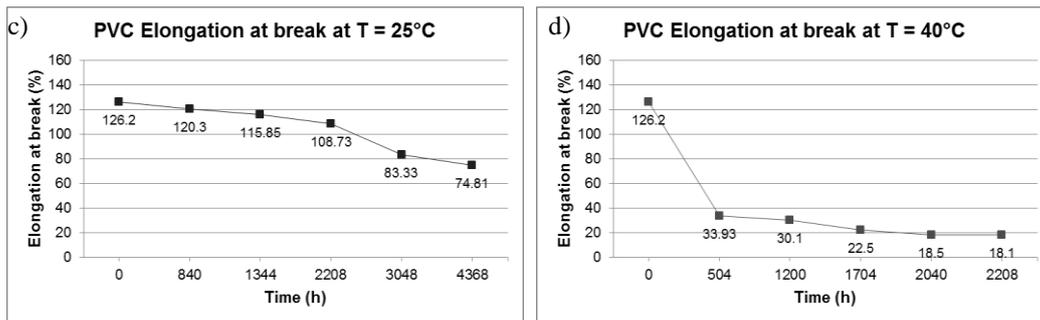


Fig. 3 PVC samples aging in sulfuric acid solution : Tensile strength as function of time at : a) 25°C, b) 40°C and elongation at break as function of time at: c) 25°C, d) 40°C.

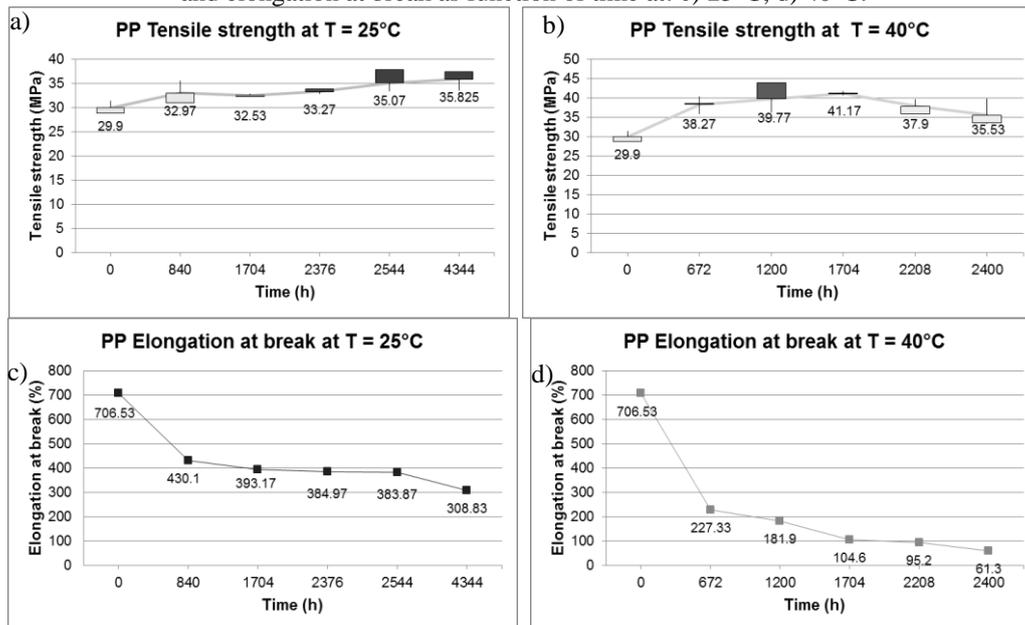


Fig. 4 PP samples aging in sulfuric acid solution : Tensile strength as function of time at: a) 25°C, b) 40°C and elongation at break as function of time at: c) 25°C, d) 40°C.

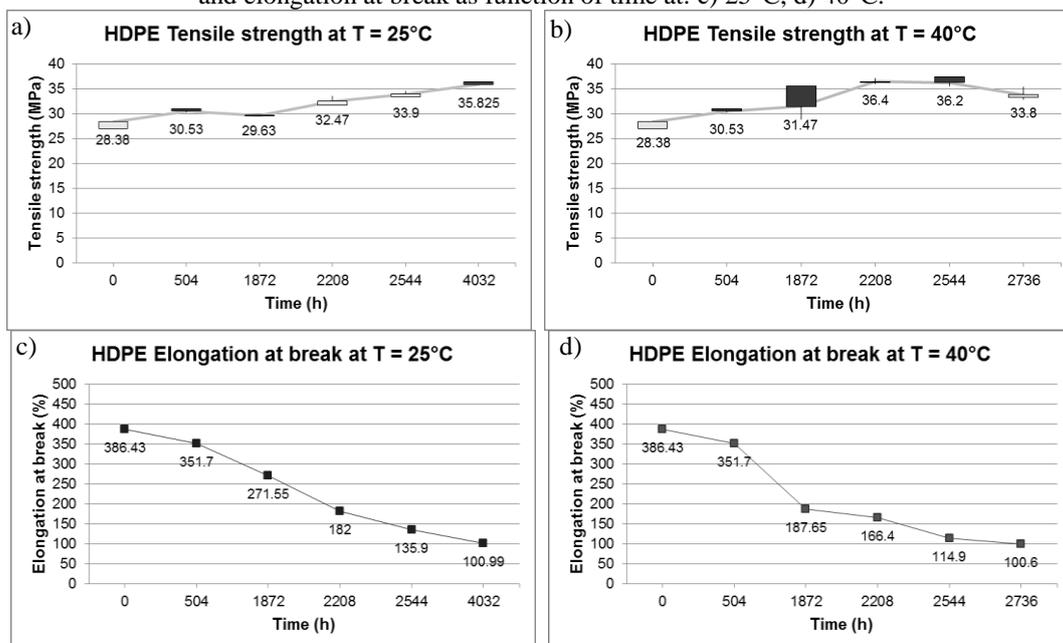


Fig. 5 HDPE samples aging in sulfuric acid solution : Tensile strength as function of time at: a) 25°C, b) 40°C and elongation at break as function of time at: c) 25°C, d) 40°C.

In conclusion, the tensile strength and the elongation at break for all materials immersed in sulfuric acid solution are higher and lower, respectively, than the unaged sample with the augmentation of time and test temperature. These results can be explained in general, by the increase of crystallinity, followed by the increase of crosslinking density due to a thermal effect, the increase of the time of exposure and the diffusion of the solvent into the materials [1-2,12-14,21-24].

As reported by [1-2,11-14,21-25], several factors may have an effect on the mechanical behavior. According to Rowe et al. [11] the increase in tensile strength for HDPE geomembrane at elevated temperature is due to the increase in crystallinity.

Kieran Murray et al. [12] show that the decrease of the mechanical properties resulted in the deterioration of the polyethylene chains. Mendes et al. [13] prove that the increase in tensile strength and the decrease in elongation of polyethylene are indicating the occurrence of two competing degradation mechanisms: scission and crosslinking. Weon et al. [14] indicate that on the molecular chain at the level of polymers, the increase in tensile strength at elevated temperature is due to the increase in chain mobility. This can lead to an increase of the chain alignments and of the lamellar thickness. In addition, the formation of large numbers of van der Waals inter-chain bonds lead to significant intermolecular forces, even if this secondary intermolecular bonds are much weaker than the primary covalent ones. In the other hand Weon et al. [14] show that the decrease in mechanical properties is due to the increase of crosslinking density. The rise in temperature and the diffusion of the solvent into the polymer, leads to the process of inter-crosslinking of the molecular chains, resulting in a chemical degradation. The crosslinking of inter-chains improve the strength by restricting the chain motions, while on the other hand it causes harmful effects on the mechanical properties due to chain scission. As reported also by [14] the decrease in elongation at break is due to the reduction of chain segments caused by molecular chain scission, of density of chain entanglement, of chain mobility and in elasticity of the aged materials. Furthermore, as reported by Bouguedad et al. [21] the decrease in mechanical properties is due to the phenomenon of chain scissions, which causes a decrease in the molecular weight and in the degree of crosslinking on the one hand and a loss of plasticizers in the other hand. Nandakumar et al. [22] show that the decrease in mechanical properties of ethylene-propylene-diene (5-ethylidene-2-norbornene) terpolymer-BaSO₄ nano composites is due to increase in crosslink density, the restriction of the mobility of the chain, which limits the orientation of the network chains. Whelton et al. [23] show that the ductile failure for HDPE samples

aged in chlorinated water at different temperatures can be attributed to chain fracture as well as chain disentanglement, which were caused by diffusion of chlorinated water induced oxidation. As reported by Mkacher et al. [24] there is a direct link between the chemical evolution of the material and its elongation at break due to the chains scissions, rather than its tensile strength which should vary in the way non-monotonously. According to Abbès et al. [25] the diffusion of solvent as well as the temperature during contacts influences the mechanical behaviour of the polymer. The temperature increases the movements of the amorphous part of PP and the diffusion of the solvent which reduces the mechanical properties.

IV. Conclusion

The present study concerning diffusion of solvent process and its effect on the mechanical properties of materials used in sewer systems (PVC, PP and HDPE), has led to the following conclusions: Gravimetric measurements show that the PVC has a fickian behavior, characterized by:

- A first portion corresponding to a rising phase of water absorption, where the weight of the sample is increasing, which indicates a penetration of water into the amorphous parts of the material to saturation.
- A second part is relatively stable, what characterizes a 'pseudo plate' of diffusion.

Gravimetric measurements show that the PP has a non-fickian behavior, characterized by a rapid acceleration of the water absorption, which is usually accompanied by large deformations and damage within the material. Also, we find that the absorption increases with rising temperature.

Gravimetric measurements show that the HDPE has a fickian behavior at 25°C, while it has a non-fickian behavior at 40°C, characterized by a descending phase, where the weight of the sample decreases gradually and becomes negative after a certain time of aging, indicating an internal reorganization and leaching of certain low molecular weight components of the material during aging process.

The sulfuric acid effect on mechanical properties, shows that the tensile strength and elongation at break for all materials immersed in sulfuric acid solution are higher and lower, respectively, than the unaged sample with augmentation of time and test temperature. These results can be explained by the increase of crystallinity, followed by the increase of crosslinking density due to the diffusion of the solvent into the materials, thermal effect and the increase of the time of exposure.

These results prove that there is a correlation between the diffusion of the solvent as well as the increase of the time of exposure and the temperature on the mechanical properties for all materials.

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