What Is The Risk Of Plastic Pipe Long-Term Degradation On Water Quality?

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Abstract

Plastic piping products are not affected by electrochemical corrosion. However, they can be severely degraded after long-term service by oxidation process notably in presence of chlorinated disinfectant. This study was conducted in order to investigate the impact of plastic pipe degradation on water quality. Polyethylene (PE) connections, with various service times from water networks disinfected by chlorine and chlorine dioxide were collected. In parallel, new pipes made with PE, cross-linked PE and unplasticized polyvinyl chloride, were aged in an accelerated manner using an ageing bench. Results confirmed PE and cross-linked PE pipe degradation by oxidation following their exposure to disinfectant notably chlorine dioxide. Testing performed on pipes aged on the bench showed that water quality can be severely impacted in certain circumstances following polyolefin pipe degradation. However, testing performed on in-situ pipes did not highlight a link between the deterioration of water organoleptic properties and pipe degradation level. Lastly, waters in contact with all plastic pipes contained few organic compounds notably antioxidant by-products.

Keywords

Ageing, Disinfectant, Drinking Water, Organic Pollutant, Plastic Pipe, Taste-and-Odor

I. INTRODUCTION

Plastic piping products, and notably polyethylene (PE) pipes, are widely used in water distribution networks especially for connections. This interest can be explained by the numerous advantages they offer (flexibility, lightness, metal corrosion free…). Plastic pipes made of HDPE, cross-linked polyethylene (PE-X) or polyvinyl chloride (PVC) are also frequently used inside buildings instead of “traditional” metallic products.

Studies have shown that the migration in water of organic substances from newly installed plastic pipes can alter drinking water odor and flavor. For PE pipes, Anselme et al. (1985) were among the earliest to report the detection of numerous organic compounds associated with taste and odor issue in drinking water. This study permitted to identify some antioxidant release but also leaching of aldehydes and ketones. The release of these latter compounds was attributed to a strong oxidation of the PE pipe inner wall during the extrusion process. Since then, many researches were performed on the release of organic compound in drinking water from plastic pipes and consequences for water quality (Rigal and Danjou (1999), Brocca et al. (2002), Rigal et al. (2003), Skjevrak et al. (2003), Marchesan and Morran (2004), Schweitzer et al. (2004), Durand and Dietrich (2007), Heim and Dietrich (2007)). Lately, Denberg (2009) has performed a review of organic species released from PE pipes in drinking water. Within the hundreds different organic compounds reported in the literature, Denberg (2009) categorizes the species into three groups of migrants: additives such as antioxidants, degradation products formed by radical reaction of antioxidants and degradation products formed by PE chains oxidative degradation.

To understand why these organic compounds can leach it is necessary to consider the basic auto-oxidation scheme of PE material. This complex radical reaction scheme is now well described in literature notably by Al-Malaika (2003). The PE auto-oxidation process is initiated by the breaking of C-H bond. Parameters such as heat light, impurities (catalytic residues) can initiate labile hydrogen detachment. In presence of oxygen the oxidation conducts to random location of chain breaking followed by formation of aldehydes, ketone, alcohol and carboxylic acids. Typically, the pipe extrusion is a potential step for initiating the oxidation. In order to avoid auto-oxidation reactions stabilizers are added to the polymer.

As highlighted by Brocca et al. (2002), some of the organic compounds released in water could be impurities presents in the stabilizers “masterbatch” added in the pure PE resin before its extrusion. Therefore, depending on PE resin brand and pipe manufacturing process, many types of organic compounds can be present in the polymer and potentially released in the water. It has been shown that many of the organic compounds leaching from plastic pipes can alter drinking water flavor. Villberg and Veijanen (1998) identified several organic compounds that contributed to modify water flavor such as C_6-C_9 ketones, toluene, C_9-C_{10} aldehydes with various flavor descriptors such as plastic, bitter, sweet, burnt plastic, model glue. Heim and Dietrich (2007) also attributed the plastic odor of PE leachate to several ketones, alkanes and phenol detected.

Research for identifying the potential impact of PE oxidation on food packaging behavior leads to similar conclusion. Andersson et al. (2005) suggested that the highest correlation between off-flavor in water packed in PE film and oxidation was found for ketones in the range of C_7-C_9 and aldehydes in the range of C_6-C_9.

Another concern about the use of PE pipes in drinking water has emerged on the last

decade when several researches showed that the long-term reliability of PE pipe systems was affected by chlorinated disinfectants and notably chlorine dioxide. Dear and Mason (2001) found that the presence of chlorine in the water accelerated the PE oxidation inducing an embrittlement of the pipe inner wall. Hassinen et al. (2004) observed a rapid chemical consumption of stabilizers by the chlorinated water and extensive polymer degradation confined to pipe inner wall. Colin et al. (2009) showed that chlorine dioxide (ClO_2) initiated the PE oxidation by breaking C-H bond. In contact with ClO_2, auto-oxidation of PE material was accelerated inducing PE chain breaking followed by formation of oxidation by-products.

These results raise the question about the impact on water quality of oxidation by-products leaching from plastic pipes after long-term ageing in chlorinated media. Few works deal with aesthetic water quality associated with plastic pipes oxidation following their exposure to disinfectant. Durand (2005) exposed PE pipes during more than 150 days to chlorinated water (1 and 4 mg/L of free chlorine). By performing flavor profile analysis (FPA) after different ageing periods, it has been highlighted a decrease of the FPA intensity with time of exposure. The study demonstrated, as it is commonly accepted, that new PE material generates odors during the first days of service but gradually decreased over time. However, Durand (2005) performed the ageing at room temperature limiting the degradation and unfortunately did not evaluate PE oxidation.

Therefore the present study attempted to assess the impact of long-term plastic pipe oxidation on water flavor and migration of organic compounds by: testing samples of in-service PE pipes (natural ageing) and accelerated aged plastic pipes.

II. MATERIALS AND METHODS

2.1 Accelerated aging experiments

The accelerated ageing experiments were conducted on 2 high-density PE (named as PEHD A and PEHD B), 1 PE-Xa (cross linking using peroxide) and 1 unplasticized PVC pipe products; all commercially available. The four pipe materials had an “Attestion de Conformité Sanitaire” (ACS) allowing their use for drinking water distribution in accordance with the French regulation. All the tested pipes had an external diameter of 25.0 mm and thicknesses of 3.0 mm, 2.8 mm and 2.3 mm respectively for PE, PVC and PE-X pipes.

Plastic pipes were exposed during 12 months to medium mineralized water heated at 40℃ with a pH maintained at 7.2. Three different d isinfection conditions were used: 4 mg/L of free chlorine, 1 mg/L of ClO_2 and no disinfectant. The water pressure was 6 bar and a water flow of 200 L/h. Previous research demonstrated that these accelerated ageing conditions allowed to simulate natural aging process and to benchmark materials according to their disinfectant resistance Rabaud and Rozental-Evesque (2008).

2.2. Natural aged pipes

10 in-service PE connection pipes were taken from two different water distribution networks. 5 PE pipes were exposed during 1 to 11.5 years to water disinfected by chlorine. 5 PE were exposed during 3.5 to 23.5 years to water disinfected by ClO_2. All the tested pipes had an external diameter of 25.0 mm and thicknesses from 2.3 to 3.0 mm offering the same surface/volume ratios as accelerated aged pipes.

2.3. Analysis of plastic pipe degradation

Oxidation Induction Time (OIT)

OIT is widely used for the determination of thermal oxidative resistance of PE materials. It is known that a linear relationship exists between the concentration of phenolic antioxidant (and also phenolic/phosphate mixtures) and the observed OIT in polyethylene. The polyolefin pipes (PE, PE-X) samples were tested at the inner and middle surface. The OIT was conducted in accordance with ISO TR 10837:1991. The test was performed at 200℃. An OIT less than 5 min suggests that no antioxidants remain in the sample.

Fourier Transform Infra-Red Spectroscopy (FTIR)

The formation of carbonyl oxidative products is one of the most common degradation mechanisms and can be identified by FTIR-ATR with their specific infrared band in the region 1710-1740 cm^{-1}. The absorbance maximum around 1720 cm^{-1} was taken as a measure of the concentration of carbonyl compounds. The Carbonyl Index (CI) for polyolefin material (PE, PE-X) was defined as the ratio of the peak height at 1720 cm^{-1} to the peak height at 1470 cm^{-1} and for PVC materials as the ratio of the peak height at 1720 cm^{-1} to the peak height at 1253 cm^{-1}.

2.4. Analysis of migration water

Migration/leaching protocol

Migration tests were performed in accordance with the French standard for testing materials intended for contact with DW XP P41-250 –1 and –2 with the difference that only pipe inner wall was in contact with migration water. Thus, given pipe dimensions, the surface/volume ratio was an average of 2000 cm^2/L. Pipes were filled several times with test waters (Ultra Pure or Evian) containing 50 mg/L or 1 mg/L of free chlorine.

Total Organic Content analysis (TOC)

The determination of TOC was performed in accordance with NF EN 1484 standard using UV / persulfate oxidation method. CO_2 was measured using Infrared analyze method.

Chlorine demand analysis

Chlorine demand were performed in accordance with NF EN ISO 7393-2 standard using colorimetric method with N,N-diethyl-1, 4-phenylenediamine). A spectrophotometer operating at a wavelength of 540nm was used for determining free chlorine concentration. 50 mg/L of free chlorine were added to migration waters for 24 h of stagnation.

Flavor assessment (TFN)

Flavors of migration waters were performed in accordance with NF EN 1622 standard protocol by applying paired test and unforced choice of the full method. Migration water were equilibrated at 23℃ and flavor assessed by at least 3 trained panelists.

Threshold Flavor Number (TFN) was determined for a maximum of seven successive stagnation periods of 24h. TFN value corresponds to the degree of dilution required to produce sample of barely perceptible flavor compared to the reference water.

GC/MS analysis

GC/MS analyses were performed in accordance with XP P 41-250-2. The identification of organic compounds in migration waters was carried out after liquid-liquid extraction using dichloromethane.

III. RESULTS AND DISCUSSION

3.1. Degradation of plastic pipes after natural and accelerated aging

Accelerated aging results

After 6 months of exposure to non-chlorinated water at 40°C, polyolefin pipes did not show any sign of oxidation (Figure 1). A slight decrease of the OIT was measured at pipe inner wall but CI indicating that no oxidation (i.e. carbonyl formation) occurred. After 6 months of chlorine exposure a significant decrease of the OIT value was highlighted. PEHD B and PE-X materials showed a decrease of about 30 % the OIT whereas PEHD A showed a decrease of more than 75 %. This latter one demonstrated also a CI value higher than 0.05 indicating an oxidation of the material. After 6 months of exposure to ClO_2 PE/PE-X pipes exhibited a dramatic drop in OIT at the bore surface suggesting a significant consumption of antioxidants. OIT measured at the middle wall (not shown Figure 1) did not demonstrate any variation. CI results for PE/PE-X pipes exposed to ClO_2 highlighted a significant level of oxidation of the pipe inner wall. PE-X pipe showed the highest level of oxidation. For PVC pipes, no significant level of oxidation was observed according to CI value whatever the aging condition (data not shown). Therefore, results showed that PE and PE-X pipes suffered oxidation due to their exposure to disinfectant. ClO_2 induced the strongest degradation.

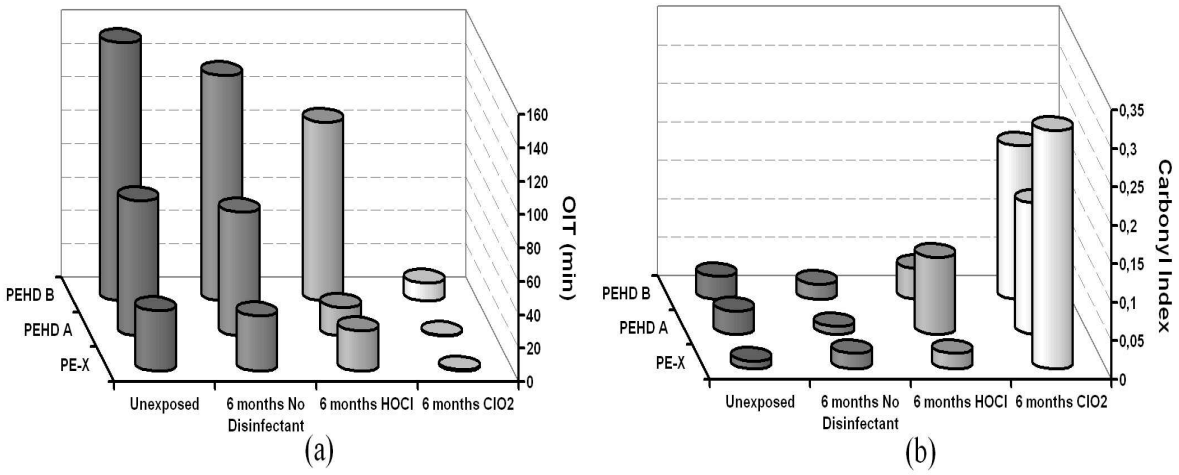
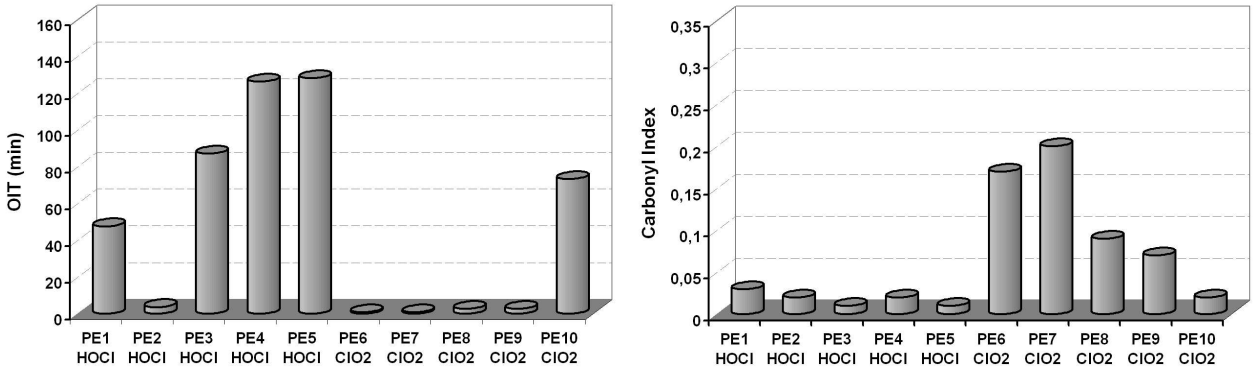


Figure 1: Oxidation Induction Time (a) and Carbonyl Index (b) measured on pipe inner wall for polyolefin pipes (PE and PE-X) before and after 6 months of accelerated aging with exposure to chlorine, chlorine dioxide and non-chlorinated water.

Natural aging results

Natural aged PE pipes exposed to chlorine did not exhibit significant sign of degradation (Figure 2). Only PE2 with the highest service time (11.5 years) demonstrated a very low OIT level. However no oxidation was noticeable according to CI value. For PE pipes exposed to water containing ClO_2, various degradation levels were highlighted. No significant loss of antioxidant and no oxidation were noticed for PE10 with the lowest service time (3.5 years). On the other hand, for PE6 and PE7 with respectively 21.5 and 23.5 years of service no residual antioxidant was found at pipe inner wall and CI level indicated a severe oxidation. It should be noted that for these two pipes, the OIT levels at the middle were greater than 10 minutes and no oxidation was noticed. The two last PE pipes with 10 years of service had no antioxidant at inner wall and exhibited a limited oxidation. Thus, natural aged pipes demonstrated similar degradation behaviour compared to accelerated aged pipes notably for ClO_2 exposure. The longer the service time was, the greater the oxidation was. And for constant service time, pipes exposed to chlorine demonstrated a lower degradation.



(a) (b)

Figure 2: Oxidation Induction Time (a) and Carbonyl Index (b) measured on PE pipe inner wall after natural aging. PE1 to PE5 were exposed to water containing chlorine. PE6 to PE10 were exposed to chlorine dioxide.

3.2. Assessment of water quality after exposure to aged plastic pipes

Accelerated aging results

TOC content of migration water in contact with unexposed plastic pipes remained at low level (0.3 mg C/L) except for PE-X material (0.9 mg C/L) (Figure 3). After the exposure to chlorinated waters (chlorine and ClO_2), no significant variation in TOC was noticed except for PE-X. In this latter case the TOC content decreased.

These results suggest that the degradation of PE and PE-X inner wall did not contribute to modify the TOC content. Only pipes exposed to non-chlorinated condition showed a significant increase of the TOC especially for PE and PE-X materials. This result can be attributed to biofilm development on the pipe inner wall during the 12 months aging with a non-disinfected water at 40℃. TOC values for non-disinfected condition also suggested that biofilm growth was slower on PVC compared to PE and PE-X. This conclusion is consistent with chlorine demand results. Chlorine demands (for 50 mg/L of chlorine) were closed to 100% for polyolefin materials. Results also highlighted that PE and PE-X materials exposed to

ClO_2 had an unexpected high chlorine demand (from 29% to 36%). This point will be discussed later. For pipes exposed to chlorine during 12 months no significant variation of chlorine demand was noticed.

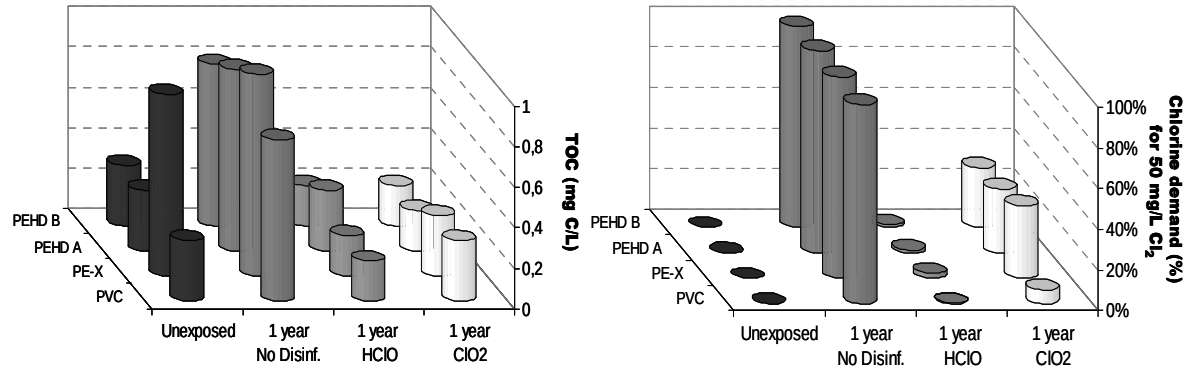


Figure 3: Total Organic Carbon (a) and Chlorine demand (b) measured for migration waters in contact with pipe inner wall before and after 1 year of accelerated aging with exposure to chlorine, chlorine dioxide and non-chlorinated water.

After the first contact period, TFN values for new pipes were greater than one indicating that the plastic pipes induced off-flavor to the migration water (Figure 4). Flavors were comparable to solvent, rotten fruit, “English candy”, astringent, bitter for polyolefin pipes and solvent, plastic and astringent for PVC pipe. TFN values decreased rapidly except for PEHD A. TFN level 3 was reached after the seventh and last contact period. For pipes exposed to non-disinfected water, the TFN values after the first contact period were greater than initial values for unexposed pipes. Only PVC pipe reached the TFN level 3. For all materials, flavors were described as plastic, astringent and rancid. This latter flavor descriptor was attributed to biofilm development on pipe inner wall. Now when considering pipes exposed to water containing chlorine and ClO_2, flavors assessment highlighted a significant increase of TFN values for PE and PE-X materials especially after ClO_2 aging. The most striking was that TFN did not decrease with the successive contact periods for PEX materials. Plastic, solvent, astringent and celluloid were flavors descriptors obtained. Once again, PVC showed the lowest TFN values and reached level 3 at the last contact period. These results tended to indicate that the pipe inner wall oxidation of PE and PE-X induced strong off-flavor to migration water. However, given the high chlorine demand of migration waters in contact with ClO_2 exposed pipes, TFN assessment was redone on oxidized PE and PE-X pipes with and without adding chlorine during water quality testing. Migration water without chlorination exhibited after the first contact period TFN values from 2 to 5 to be compared to TFN values from 5 to 10 for water migration with chlorination. Thus, chlorination performed during water quality testing induced the appearance of off-flavors. These results suggested that oxidation of PE pipes by ClO_2 induces the formation of leaching by-products that no modify water organoleptic properties except if a re-chlorination is performed. Reaction between oxidation by-products and chlorine could be an explanation of this observation.

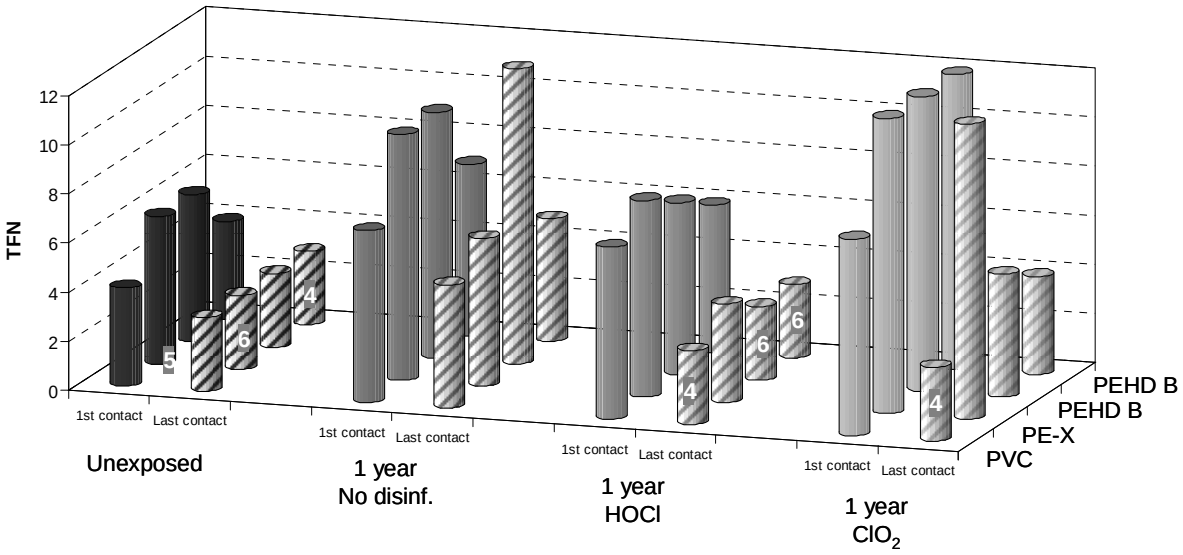


Figure 4: Threshold Flavor Number evaluated after the first contact period and the last contact period for water samples in contact with pipe inner wall before and after 1 year of accelerated aging with exposure to chlorine, chlorine dioxide and non-chlorinated water. If the last contact period was not the seventh, the number of the last contact period is indicated with the white number in a grey square.

GC/MS analysis performed on unexposed pipes revealed the presence of 7,9-di-tert-butyl-1-oxaspiro(4,5)deca-6,9-diene-2,8-dione (N°C AS 82304-66-03) for PE and PE-X materials. This compound was previously identified by Brocca et al. (2002) and was classified as a phenolic antioxidant by-product. PEHD B exhibited the highest relative concentration of the antioxidant by-product. For PVC material no specific organic compound was identified. After 6 months of aging, whatever the disinfection conditions, the relative concentration of anti-oxidant by-product tended to decrease. After 6 months of ClO_2 aging the antioxidant by-product was identified only for PEHD B material. Based on the first GC/MS results, no correlation between the high TFN levels and organic compound release was highlighted.

Natural aging results

Migration waters exposed to natural aged pipes demonstrated a TOC content equal or lower than 0,2 mg C/L. The chlorine demands (for 50 mg/L of chlorine) were between 0 and 7% both for pipes exposed to chlorine or ClO_2. Thus, the high chlorine demands highlighted for PE pipes exposed to ClO_2 on the aging bench were not noticed for natural aged samples. TFN assessment demonstrated higher flavor intensity for PE pipes from network with ClO_2 disinfection (Figure 5). However, TFN levels for ClO_2 pipes remained much lower than those measured on accelerated aged pipes. Moreover, no correlation was found between TFN levels and pipe inner wall degradation. Thus, PE10 sample that not demonstrated oxidation showed the same TFN level than pipe with strong inner wall oxidation. GC/MS experiments allowed identifying antioxidant by-products 7,9-di-tert-butyl-1-oxaspiro(4,5)deca-6,9-diene-2,8-dione and 2,6-di-tert-butyl-p-benzoquinone in the migration waters for all PE pipes. PE pipes exposed to chlorine demonstrated higher relative concentration

of these two compounds with the highest concentration for pipes with the lowest service time. GC/MS analysis also showed traces of phthalate compounds. Thus, despite high oxidation levels highlighted for some ClO_2 natural aged pipes, results obtained did not show trend of water quality degradation due to PE pipe oxidation. These results are different from those obtained with accelerated aging samples despite the fact that oxidation of pipe inner wall was noted in both cases. However, it can be excluded that during the accelerated aging there was a concentration of leachable compounds at pipe inner wall. During, natural ageing due to the slowness of oxidation process leaching of organic compounds inducing off-flavor could be more scattered.

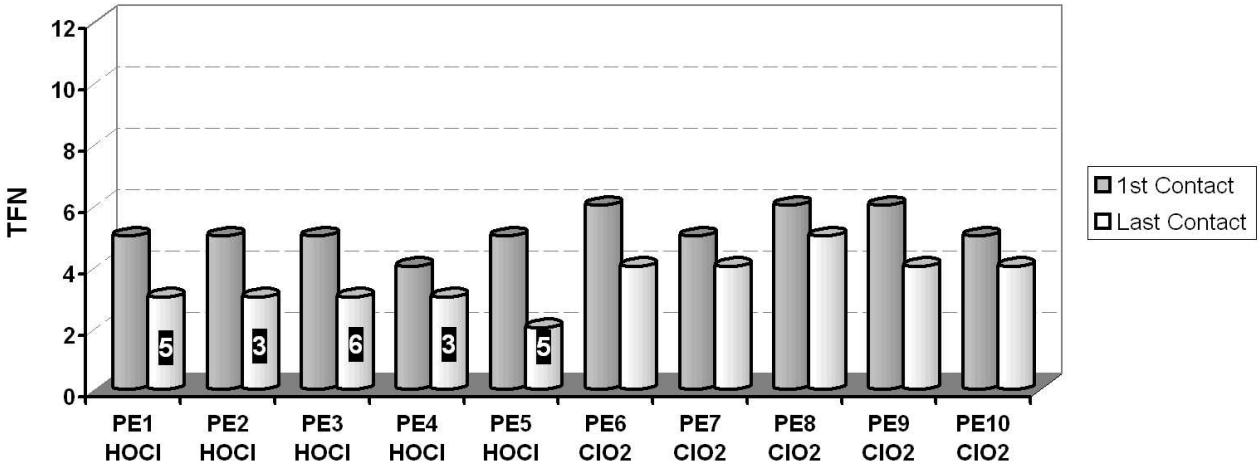


Figure 5: Threshold Flavor Number evaluated after the first contact period and the last contact period for water samples in contact with PE pipes after natural aging. PE1 to PE5 were exposed to water containing chlorine. PE6 to PE10 were exposed to chlorine dioxide. If the last contact period was not the seventh, the number of the last contact period is indicated with the white number in a black square.

IV. CONCLUSIONS

- Chlorine and ClO_2 induced premature degradation of the PE and PE-X pipe inner wall. Exposure to disinfectant induced a consumption of the antioxidants followed by an oxidation of pipe inner wall. ClO_2 is much more aggressive than chlorine. These results were obtained both on accelerated aged and natural aged pipes. PVC pipes did not demonstrate oxidation after exposure to chlorine or ClO_2.
- Oxidized pipes did not impact the TOC content of migration waters. For accelerated aged pipes, oxidation due to ClO_2 led to a significant increase of chlorine demand of migration water. Results suggested that the chlorine demand was due to the reaction of chlorine added during water quality testing and oxidation by-products formed during PE degradation by ClO_2.
- Sensory assessment of migration waters revealed some off-flavors with descriptors such as plastic, astringent, solvent, rotten fruit and celluloid. PE and PE-X pipes demonstrated higher flavors levels than PVC pipe regardless degradation levels.
- Migration waters from polyolefin pipes accelerated aged and exposed to ClO_2 demonstrated very strong off-flavors only when chlorine was added in the test

water. With non chlorinated test waters, the level of flavor (TFN) is much lower. This suggested that oxidation of PE pipes by ClO_2 induces the formation of by-products that do no affect water flavor except if a re-chlorination is performed.

- For in-service pipes, despite the relative high oxidation levels highlighted for some ClO_2 natural aged pipes, results obtained did not show trend of water quality degradation due to PE pipe oxidation despite the fact that migration water exhibited off-flavors.

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NF EN ISO 7393-2 Qualité de l'eau - Dosage du chlore libre et du chlore total - Partie 2: méthode colorimétrique à la N, N-diéthylphénylène-1,4 diamine destinée aux contrôles de routine.

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