**CIVIL & ENVIRONMENTAL ENGINEERING | RESEARCH ARTICLE**

# Durability study of reinforced polyester composite used as pipe lining under artificial aging conditions

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**Abstract:** The aging of sewer infrastructure is an ongoing problem. As a result, different materials and methods are being used in alternative sewer rehabilitation approaches. This work was conducted to study one pipe lining, namely the reinforced polyester composite, under artificial aging; this was done to provide a better understanding of the material's performance under operating conditions, where it is regularly exposed to degrading factors such as heat and water. Aging of the material was monitored by means of several tests, including thermal and mechanical analyses, water absorption and microscopy. The results showed that the combination of aging in water and at high temperatures resulted in greater effects on the material compared to aging at high temperatures in dry conditions. Although the measured properties were affected significantly when immersed in water at high temperatures, the material showed acceptable properties at lower exposure temperatures close to the expected temperature inside sewer systems.

**Subjects: Coatings & Thin Films-Materials Science; Composites; Corrosion-Materials Science; Polymers & Plastics; Surface Engineering-Materials Science; Polymer Technology; Civil,Environmental and Geotechnical Engineering; Cities & Infrastructure**

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Parastou Kharazmi holds a Master as well as a PhD degree from the KTH Royal Institute of Technology in Stockholm, Sweden. She has ten years working experience as researcher, technical advisor and specialist in different industries and her current research focuses on application of polymeric composite materials for sustainable buildings and infrastructure maintenance.

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## PUBLIC INTEREST STATEMENT

Water and wastewater piping systems are among the costliest infrastructure investments. Polymeric lining can be used to eliminate deterioration of the pipeline and thereby extend the service life. Although rehabilitation technologies employing polymeric systems have been in use for over 30 years, there have been few technical assessments of these technologies or the materials involved. The main objective of this study was to contribute to an improved understanding of one of the commonly used pipelining materials, polyester-based lining.

Degradation of the material was investigated via artificial aging involving immersion in water at elevated temperatures. The changes in materials that occurred during accelerated aging were tracked by means of various tests. Results indicated that the properties of the material changed significantly when high temperatures were combined with water exposure. However, the aging testing also found that the material performed relatively well at temperatures close to the average temperatures inside sewer systems.

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## 1. Introduction

The rehabilitation of aging pipes and sewers is one of the most costly types of infrastructure repair. As a consequence, the use of alternative rehabilitation methods, which are faster and more economical compared to traditional pipe replacement, has increased significantly in recent years. One of these methods—so-called relining or pipe linings—involves the application of a polymer coating on the inner surface of the pipe to form a rigid protective liner inside the deteriorated pipe after curing. The polymers used for this type of rehabilitation are classified as synthesis-engineered linings (Steward, Allouche, Baumert, & Gordon, 2012). Normally, pipe lining using polymer coating is categorized as an adhered semi-structural or non-structural rehabilitation method, as it does not substantially increase the structural strength of the pipe (American Water Work Association [AWWA], 2014; US Environmental Protection Agency, 2009). The corrosion resistance liner is also categorized as a corrosion protection rehabilitation method.

Although wastewater pipe rehabilitation through the application of a polymer coating on the inner surface of the existing pipe has been conducted for over 30 years, very few quantitative studies have evaluated the performance of the materials used in these emerging innovative technologies (Selvakumar, Morrison, Sangster, & Condit, 2013; Sterling, Wang, & Morrison, 2009).

The objective of this work was to study one of the lining materials, regularly used in Sweden for rehabilitation of small diameter pipes in residential buildings in Sweden, which is a glass-flake-reinforced polyester polymeric system. Accordingly, this paper provides performance evaluation of this reinforced polyester *in situ* coating when it is exposed to artificial aging in order to improve understanding of the material's performance in-service conditions.

Aging of the material was studied in the lab by monitoring changes in the material when it was exposed to different temperatures (20℃, 40℃, 60℃ and 80℃), both when immersed in water and under dry conditions. Changes in the material during aging were monitored by conducting different tests, including thermal and mechanical analysis, water absorption and microscopy.

The polymer matrix in this lining material is primarily made from unsaturated polyester, which is further reinforced by glass-flake particles and applied via a spray-on method. Polyester resins are usually produced through the condensation of polybasic acids with polyhydric alcohols or their derivatives (Boinard, Pethrick, Dazel-Job, & Macfarlane, 2000). Although all polymers pass moisture through them, the process of modification using glass-flake particles can provide them with more resistance against corrosive factors. For application in the field, the resin is mixed with a hardener; the mixture is usually then applied in multiple layers and is thus also referred to as a "high build". Commonly, three layers are applied, with each layer being 900–1100 μm in thickness. The curing of the applied coating is expected to take 12 h at ambient temperature (Jotun, 2016).

Polymers are sensitive to aging factors, such as heat and water, which are present inside sewerage systems. During aging, deformation occurs at the molecular level, as the polymer moves to the lowest energy state available and its stiffness changes. The aging of the polymer must be considered when predicting the long-term behavior of the polymer materials in different applications (Vining, Jordan, & Hall, 2001). The aging study in this work was carried out by exposing cured sheets of material to air and water aging at different temperatures in the laboratory and monitoring the deterioration by testing the material's properties. The exposure temperatures chosen included ambient temperature and 40℃ (the expected temperatures inside the sewerage systems), as well as 60℃ and 80℃ (temperatures close to and above the glass transition temperature of the resin, at which more changes in the material are expected to occur).

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## 2. Materials and methods

### 2.1. Sample preparation
The samples studied in this paper were prepared and cured in the lab from a multi-component material including unsaturated polyester (Baltoflake Ecolite), and a catalyst (Norpol Peroxide) (Jotun, 2016).

To prepare the samples for artificial aging in the lab, the resin was mixed with 1.5 vol% catalyst at room temperature, and a 1 mm-thick layer was obtained by spreading the material over a glass plate covered with a film using an applicator. The film was cured at room temperature. After one and 2 h, respectively, the second and third layers were applied in a similar fashion. The material was stored at room temperature before being cut into pieces of different sizes; subsequently, the exposure experiments were begun.

For the aging experiments, sheets of cured material in the lab were cut and placed in jars, which were then filled with water and placed in ventilated ovens at 40°C, 60°C and 80°C or stored at room temperature (20–22°C). Half of the samples were exposed to air aging alone.

### 2.2. Testing methods
A number of different methods were used to monitor the changes in the material: these included visual inspection, optical microscopy, thermal analysis (dynamic mechanical analysis (DMA) and differential scanning calorimetry (DSC)), flexural testing and water absorption measurements.

Due to the wide variety of the aging conditions utilized, it was not possible to include all the results in this paper. Hence, only the results of some of the aging conditions and times that provide an overview of the material's behavior during aging were included.

#### 2.2.1. Microscopy
Microscopy images were obtained using a light microscopy instrument. Samples were cut from the aged sheets and interfaces of the cut prepared samples were studied under microscopy.

#### 2.2.2. Water absorption
Water sorption measurements in this study were carried out by immersion of material samples in water at different temperatures (20°C, 40°C, 60°C and 80°C). The results of the sorption or weight change ratio, $W (\%) = (w_2 - w_1) / w_1) * 100$ (here, $W$ is the weight change, $w_1$ is the weight before immersion and $w_2$ is the weight after immersion), were studied by means of curves where the weight change percentage was plotted against exposure time (hr) or normalized time ($Time^{1/2}/Thickness of the sample (hrs^{1/2}/mm)$).

#### 2.2.3. Thermal (Tg) analysis
The glass transition temperature (Tg) is the temperature range in which the resin transforms from a hard, glassy solid to a viscous liquid (2000) and is an indication of changes in the material and its chemistry. Studying Tg can assist in identifying changes in the chemistry of certain materials. In this study, Tg measurements obtained through differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA) were used to track changes in the material's properties when it is exposed to degrading factors during artificial aging.

##### 2.2.3.1. DSC.
DSC measurements were performed using a Mettler instrument. Samples of each material (approximately 10 mg) were first heated in 40 µl aluminum pans from −20°C to 180°C. For the second scan, the samples were cooled from 180°C to −20°C; in the last scan, the samples were again heated to 180°C. For all scans, the samples were held for 5 min at −20°C and at 180°C. Tg can be determined by measuring the midpoint from the heating phases, in accordance with ASTM E1356 (American Society for Testing and Materials [ASTM], 2014).

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**2.2.3.2. DMA.** DMA measurements were performed using a Perkin Elmer instrument (2000) with a single cantilever beam. The temperature was scanned at a constant heating rate (5°C/min) and a constant frequency (1 Hz) from −20°C to 180°C in a liquid nitrogen atmosphere. Tg was determined according to ASTM D7028 (ASTM, 2015) from the peaks in the curve of tan delta plotted against temperature.

### 2.2.4. Flexural testing

Mechanical analysis is the most fundamental testing to study materials' properties. Mechanical testing in this work took the form of flexural testing (three-point bending) according to International Standards, ISO 178 (2001); this is a test method used for the determination of flexural properties, including flexural modulus and flexural strength. An Intron tensile machine equipped with a three-point bending fixture was used. The cross-head speed was 2 mm/min, and the load cell was 500 N. Test specimens were cut from the aged liner sheets in the shape of a beam and the dimensions were entered manually into the machine. Testing was performed at room temperature.

## 3. Results and discussion

### 3.1. Water absorption

Water sorption measurements were carried out by immersing coupons of material in water at different temperatures (20°C, 40°C, 60°C and 80°C). Comparison of the initial water uptake of samples at different temperatures demonstrates that the amount and rate of water uptake increases as the exposure temperature increases; see Figure 1. One reason for the degradation of the material is the hydrolysis of the resin, which causes a reduction in the density of cross-linking and consequently more water permeability (Boinard et al., 2000). Another reason is that debonding between the resin and glass-flake particles occurs at higher temperatures, which permits more water uptake. Higher temperatures (60°C and above) can cause consistent degradation in a polymeric system based on polyester resin (Carra & Carvelli, 2015).

The material showed increases in weight of up to 4.44% during 2 months of immersion before eventually reaching equilibrium, as seen in Figure 2. Continued measurement up to 14 months showed a far reduced water uptake, as seen in Figure 3. Reduction of water absorption after reaching equilibrium is a sign of the material's degradation. Relatively low and slow water uptake during water immersion is a sign of the strong inner bond in the composite in general, as well as the material's increased stability against degrading factors.

### 3.2. Thermal analysis

Tg measured via DMA increased as the exposure temperature increased from 20°C to 60°C, but then decreased at 80°C, as can be seen in Figure 4. Considering that the mechanism of the Tg

**Figure 1. Water uptake (Weight change (%)) for samples immersed in deionized water during the initial 2 weeks of exposure time.**

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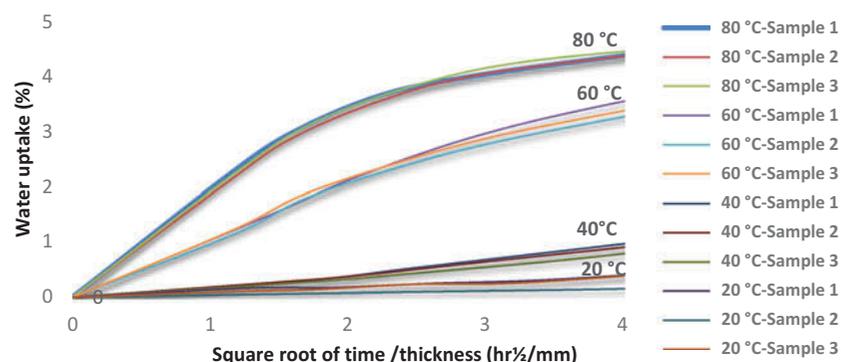
**Figure 2. Water absorption plotted as weight gain ratio against time for a coupon exposed at 60°C for 2 months.**
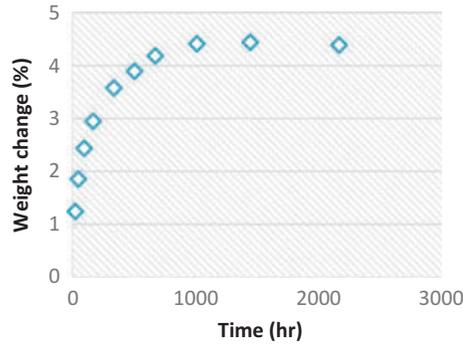


**Figure 3. Water absorption curve for a coupon exposed at 60°C for 14 months.**
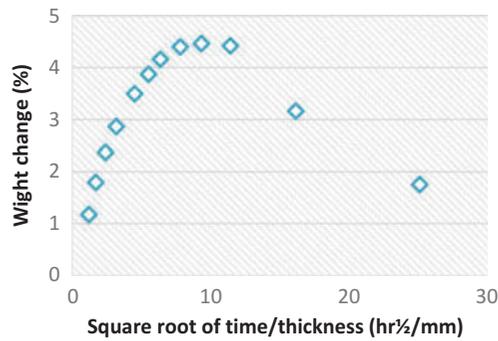


**Figure 4. Tg measurements via DMA for samples after water aging for 2 months at room temperature (20 −22°C), 40°C, 60°C and 80°C and drying.**
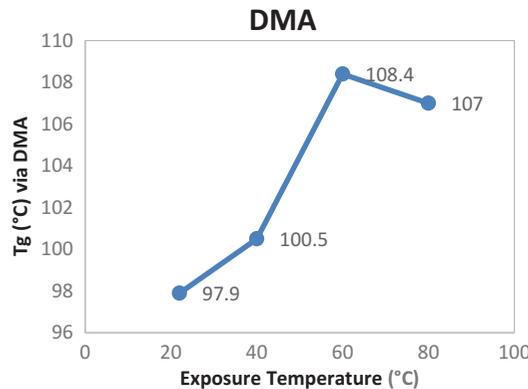


measurement used in DMA is a mechanical shock, a decrease in Tg at 80°C was expected, as it was observed that the material's properties generally change due to degradation at this temperature. Analysis of the DSC measurements revealed similar behavior on part of the material as was identified through the DMA measurements, with an increase in Tg (from the second heating phase in DSC) from room temperature (20°C-22°C) up to 60°C and then a decrease at 80°C, as seen in Figure 5. The different Tg values observed from the DSC and DMA measurements arose because the measurement mechanisms differ between these two instruments. It must also be noted that Tg may not be an exact point, but rather a range at which a material undergoes softening (Jotun, 2016). Since Tg highlights the temperature at which the material is stable, a decrease in Tg at 80°C observed using DSC and DMA can be a sign of deterioration of the material; this assumption is also indicated in other analyses in this study.

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**Figure 5. Tg measurements via DSC for samples after water aging at room temperature (20 −22°C), 40°C, 60°C and 80°C and drying.**
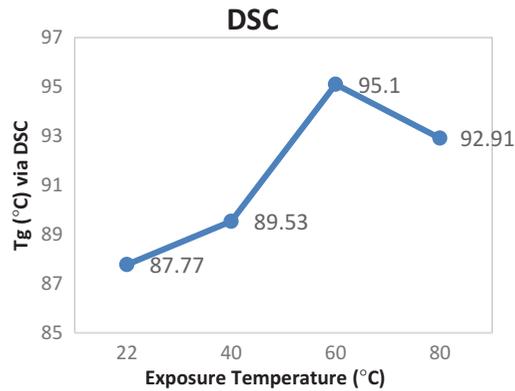


### 3.3. Flexural testing (three-point bending)

The specimens immersed in water demonstrated a continued reduction in mechanical performance compared to materials that underwent air aging, as can be seen in Table 1–4. Mechanical testing further showed that flexural modulus and strength decreased up to 57% and 58% (Table 1–2), respectively, for material immersed in water for 2 months when compared to the material exposed to similar temperatures in air aging; however, these mechanical properties also improved significantly after the aged samples were dried as shown in Figure 6–7 . Results for elongation did not differ significantly between different conditions. Moreover, the differences between the flexural modulus and strength of the materials exposed to wet and dry conditions were higher under longer exposure times and exposure to higher temperatures. It was observed that the reduction in the flexural modulus and strength after 6 months of water aging was more significant at 80°C, where the decreases were 77% and 72%, respectively, compared with the material subjected to air aging (Table 3–4). Weakness in mechanical properties can result in failures, including cracks and other damage, in the liner film. Moreover, the observed delaminations at the interface of the applied layers (Table 5) for samples that underwent water aging at 80°C demonstrated that the material would be more brittle during mechanical testing. Debonding of the interface between the glass-flake and polymer matrix can also decrease the expected reinforcement effects of the reinforcing particles (Sterling et al., 2009). Water diffusion diminishes the majority of inner-polymer bonds, while high temperatures can cause the degradation of the polyester matrix (Carra & Carvelli, 2015); both of these leads to a reduction in mechanical properties. The service life and performance of the materials in operation are highly related to the mechanical properties. When comparing the effects of aging between artificial aging and operational conditions, it should be noted that the lining material is exposed to water only from one side during operation that the average temperature inside the sewerage systems in residential buildings is known to be 40°C, and that the average day flows in water lines are highest during a few hours in the morning and evenings and very low in the late evening (Mazraeh & Abdullahi, 2018; US Environmental Protection Agency, 2002; Wallin, 2015); this underscores the fact that the lining material will not be constantly exposed to water or high temperature outside of the lab. A less significant changes in the material's properties after

| Table 1. Mechanical properties of polyester composite samples after 2 months of air aging | | | |
|---|---|---|---|
| Aging temperature (°C) | Flexural modulus (MPa) ±Standard deviation | Flexural strength (MPa) ±Standard deviation | Elongation% ±Standard deviation |
| 20 | 5289 ± 455 | 65.6 ± 7.1 | 1.4 ± 0.1 |
| 40 | 5230 ± 282 | 50.7 ± 5.6 | 1.2 ± 0.1 |
| 60 | 5792 ± 430 | 62.4 ± 4.5 | 1.1 ± 0.1 |
| 80 | 6016 ± 396 | 57.3 ± 4.7 | 1.0 ± 0.1 |

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| Table 2. Mechanical properties of polyester composite samples after 2 months of water aging | | | |
|---|---|---|---|
| Aging temperature (ºC) | Flexural modulus (MPa) ±Standard deviation | Flexural strength (MPa) ±Standard deviation | Elongation% ±Standard deviation |
| 20 | 2236 ± 249 | 37.9 ± 2.4 | 2.1 ± 0.1 |
| 40 | 2593 ± 86 | 29.2 ± 2.1 | 1.5 ± 0.1 |
| 60 | 2458 ± 161 | 26.4 ± 2.4 | 1.4 ± 0.1 |
| 80 | 3006 ± 527 | 24.6 ± 4.9 | 1.1 ± 0.2 |

**Figure 6.** Flexural modulus for samples after air aging, water aging for 2 months and drying after water aging after the same exposure time.
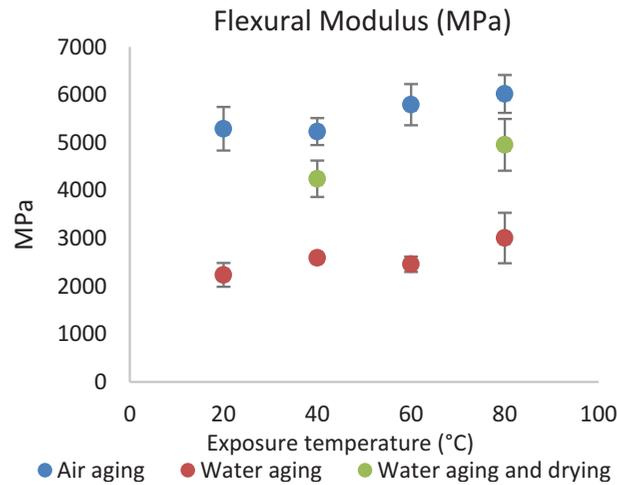


**Figure 7.** Flexural strength for samples after air aging, water aging for 2 months and drying after water aging after the same exposure time.
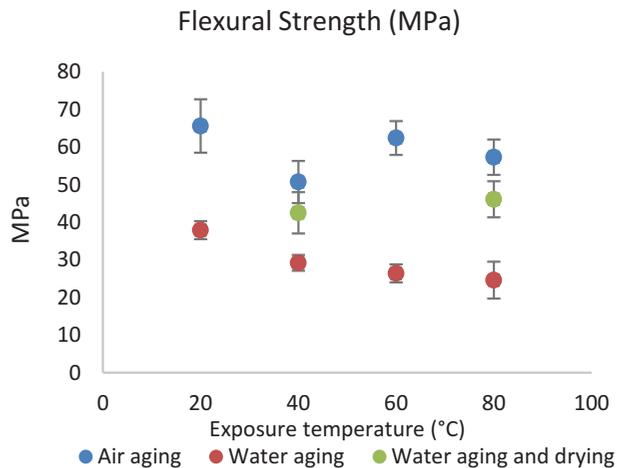


| Table 3. Mechanical properties of polyester composite samples after 6 months of air aging | | | |
|---|---|---|---|
| Aging temperature (ºC) | Flexural modulus (MPa) ±Standard deviation | Flexural strength (MPa) ±Standard deviation | Elongation% ±Standard deviation |
| 20 | 4612 ± 531 | 48.4 ± 7.7 | 1.6 ± 0.3 |
| 40 | 5720 ± 448 | 63.8 ± 4.7 | 1.4 ± 0.1 |
| 60 | 5683 ± 1151 | 57.6 ± 12.5 | 1.2 ± 0.3 |
| 80 | 5367 ± 742 | 48.9 ± 8.1 | 1.0 ± 0.2 |

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| Table 4. Mechanical properties of polyester composite samples after 6 months water aging | | | |
|---|---|---|---|
| Aging temperature (ºC) | Flexural modulus (MPa) ±Standard deviation | Flexural strength (MPa) ±Standard deviation | Elongation% ±Standard deviation |
| 20 | 2055 ± 92 | 28.9 ± 3.0 | 1.7 ± 0.2 |
| 40 | 2631 ± 313 | 33.5 ± 1.1 | 1.6 ± 0.2 |
| 60 | 2565 ± 391 | 23.6 ± 6.4 | 1.3 ± 0.2 |
| 80 | 1192 ± 479 | 13.8 ± 6.9 | 1.6 ± 0.8 |

installation in operation compared to the changes occurring during artificial aging in the lab was also confirmed in a previous study conducted by Kharazmi (2019a) in which the properties of the exposed material in real condition were tested after several years of exposure to working condition after installation.

### 3.4. Optical microscopy and visual inspection

Images 1, 2 and 3 in Table 5 show the three applied layers of the lining without any major defects after aging during 2 months at 20℃, 40℃ and 60℃ (the air bubbles were created due to aspects of the preparation in the lab, and are not expected to occur when applied by airless spray in the field). Microdelamination and delamination, as seen in Images 4 (a) and (b) (taken from samples immersed in water at 80℃) show the defects caused by degradation-related changes to the material at the highest temperatures used in this study after 8 weeks of combined exposure.

| Table 5. Optical microscopy images of the multi-layer interfaces of the cut aged samples | |
|---|---|
| Image 1: Interface of a sample after water aging for 2 months at 20℃. |  |
| Image 2: Interface of a sample after water aging for 2 months at 40℃. |  |
| Image 3: Interface of a sample after water aging for 2 months at 60℃. |  |
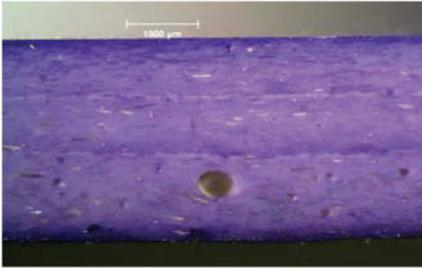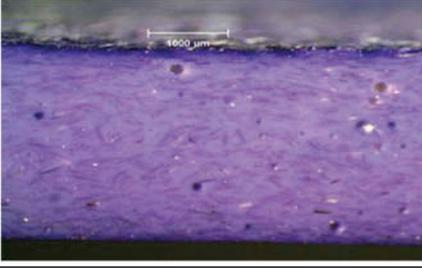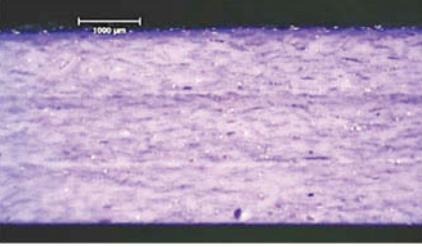
*(Continued)*

| | |
|---|---|
| Image 4 (a) and (b): Interface of a sample after water aging for 2 months at 80°C. |  |
| Image 5 (a) and (b): Interface of a sample after air aging for 2 months at 80°C. |  |
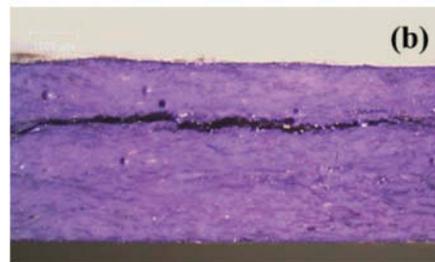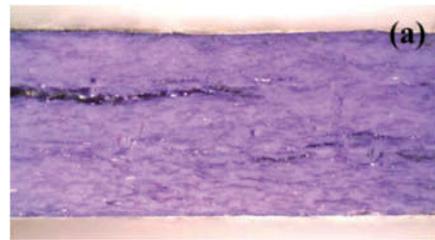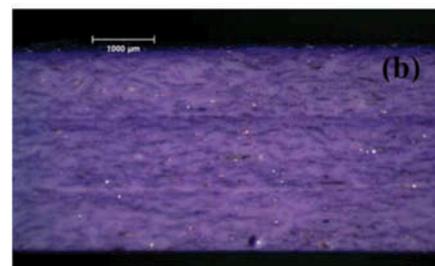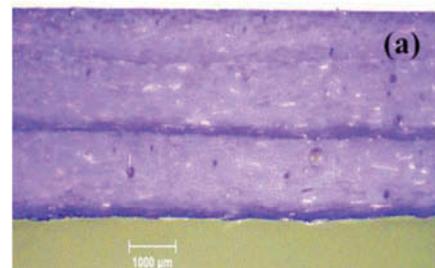
However, the sample that underwent air aging at 80°C over a similar aging time did not exhibit any major defect or delamination between the lining's layers, as seen in Images 5(a) and (b). A comparison between Images 5 (a)/(b) and 6 (a)/(b) reveals that the material is more affected by water than high temperature: while delamination and deterioration were observed for the material that underwent water aging at 80°C, the material showed more stability under air-aging conditions at a similar temperature and exposure time.

Considering that the liner material bonded to the pipe is not immersed in water during operation, but rather comes into contact with water only from one side, the constant exposure to water causing majority of the delaminations is relatively extreme. Moreover, material in the field is not exposed to water constantly at such high temperature (Mazraeh & Abdullahi, 2018; Wallin, 2015; US Environmental Protection Agency, 2002) and as previously studied by Kharazmi (2019b), the

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exposed material to real working condition, delamination issue would be minor if the material is installed properly and via a high-quality installation.

## 4. Conclusion

The polymeric systems used in the renewal of wastewater sewers in residential buildings are regularly exposed to water and heat during their service life. Thus, an aging study was conducted in the laboratory to study the weaknesses and strengths of the material so as to obtain a better understanding of its performance under operating conditions. It was observed through different analyses that the material exhibited a significant change in properties when high temperature was combined with water exposure.

Although the material's properties were affected significantly when immersed in water at high temperatures, no such major changes due to deterioration were observed at lower aging temperatures. It should be noted that the average temperature inside the sewerage system is known to be 40°C, that the material is exposed to water on only one side, intermittently, and mainly during certain hours, and that the day flow in lines for residential users is high during the early mornings but very low during late evenings. This suggests that the conditions of several months of constant water immersion or exposure to high temperatures (such as 80°C) are comparatively extreme. The results of this study showed good stability and strength for the material under less extreme conditions; therefore, it can be concluded that when the material is not continuously exposed to water and high temperatures, it is expected to perform well during its service life.

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