Assessment of Long-Term Aged Polyurethane Foam in District Heating Pipes and the Applicability of FTIR Spectroscopy

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Abstract

Polyurethane (PUR) foam used in district heating pipe systems plays a critical role in the performance and service life of the pipe assembly. Besides providing thermal insulation, the foam contributes to the structural integrity of the system by supporting the steel service pipe, maintaining the cohesion between the steel pipe and the outer casing, and limiting pipe displacement caused by thermal expansion. Over time, however, PUR foam undergoes ageing and degradation, which lead to deterioration of its thermal and mechanical properties. Fourier Transform Infrared Spectroscopy (FTIR) is commonly used to evaluate the chemical degradation of PUR by monitoring changes in molecular bonds. In this study, PUR foam samples extracted from field-aged district heating pipes at the end of their service life were investigated using FTIR and complementary analytical techniques. The outer surface of the foam exhibited strong darkening and FTIR measurements performed directly on this layer showed a significant reduction or disappearance of characteristic absorption bands. After removing approximately 1 mm of the surface layer, the typical PUR spectral features became detectable again. Additional investigations using X-ray diffraction (XRD) and chemical analysis were carried out to determine whether iron oxides or metallic ions originating from the steel service pipe contributed to this phenomenon; however, no evidence of such contamination was detected. Furthermore, nuclear magnetic resonance (NMR) analysis was conducted to examine potential hydrogen redistribution across the foam from the steel pipe side toward the casing. The results indicate that surface alterations in severely aged PUR foam may influence FTIR measurements and should be considered when interpreting spectroscopic analyses of field-aged insulation materials.

Introduction

District heating (DH) systems play a central role in modern energy infrastructure by enabling efficient heat distribution from centralized sources to consumers. Pre-insulated district heating pipes typically consist of three main components: a steel service pipe, a rigid polyurethane (PUR) foam insulation layer, and an external high-density polyethylene (HDPE) casing. The PUR foam performs multiple functions within the pipe assembly. In addition to its primary role as thermal insulation, it provides mechanical support for the steel pipe, maintains the structural cohesion between the steel pipe and the casing, and restricts pipe displacement caused by thermal expansion during operation. The long-term performance and reliability of district heating networks therefore strongly depend on the durability of the PUR insulation material (Yarahmadi, Vega, und Jakubowicz 2017). During long-term service, PUR foam is exposed to elevated temperatures, mechanical stresses, and environmental influences that gradually lead to ageing and degradation of the polymer structure. Several degradation mechanisms have been reported for PUR insulation materials in district heating systems, including thermal ageing, oxidative degradation, and gas diffusion within the foam cells. These processes may cause structural changes in the polymer network, loss of mechanical strength, increased brittleness, and deterioration of thermal insulation properties (Yarahmadi und Sällström 2014). Previous studies have demonstrated that the chemical degradation of PUR foams is largely governed by thermo-oxidative processes, which lead to changes in the molecular structure of the polymer (Yarahmadi, Vega, und Jakubowicz 2017). Accelerated ageing experiments have shown the formation of additional carbonyl groups and the reduction of methylene (CH_2) groups in the polymer backbone when PUR foams are exposed to elevated temperatures in the presence of oxygen. These chemical modifications are directly associated with the progressive deterioration of the polymer structure (Yarahmadi, Vega, und Jakubowicz

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2017).

Spectroscopic techniques, particularly Fourier Transform Infrared Spectroscopy (FTIR), are widely used to investigate the chemical degradation of polyurethane materials. FTIR enables the monitoring of characteristic absorption bands associated with urethane linkages, carbonyl groups, and other functional groups within the polymer chain, allowing researchers to track chemical changes during ageing. Several investigations of naturally and artificially aged PUR foams have demonstrated that degradation processes can be identified through systematic changes in FTIR spectra, particularly in the carbonyl and aromatic regions of the spectra (França De Sá u. a. 2017). In the context of district heating systems, FTIR has been frequently used to analyze the ageing behavior of PUR insulation materials and to correlate chemical changes with the mechanical and thermal performance of the pipes. However, the degradation process in PUR insulation is complex and may involve several phases, including early physical ageing followed by progressive chemical degradation. These processes may occur differently depending on the local thermal and mechanical conditions within the insulation layer (Alberto Vega 2018). Despite the extensive use of FTIR in polymer degradation studies, the interpretation of spectra from naturally aged materials recovered from long-term field operation remains challenging. Field-aged insulation materials may exhibit heterogeneous ageing patterns due to gradients in temperature, oxygen diffusion, and mechanical stress across the insulation layer. In district heating pipes, the most severe conditions often occur near the interface between the steel service pipe and the PUR foam, where thermal loads and mechanical stresses are highest (Weidlich u. a. 2020).

The present study investigates PUR foam insulation extracted from district heating pipes that have been in field operation for 27 years. Particular attention is given to the chemical characterization of the foam using ATR-FTIR spectroscopy and the evaluation of potential factors influencing the spectroscopic measurements. Complementary analytical techniques, including X-ray diffraction (XRD) and chemical detection methods for iron species, were employed to examine whether possible migration of corrosion products from the steel pipe could influence the spectroscopic response. In addition, nuclear magnetic resonance (NMR) analysis was conducted to explore potential changes in hydrogen distribution within the foam structure. By combining these analytical approaches, this work aims to improve the understanding of ageing phenomena in PUR insulation under real operating conditions and to evaluate potential limitations of FTIR analysis when applied to severely aged polyurethane materials from district heating systems.

Methodology

Sample Material

PUR foam samples were obtained from district heating pipe assemblies that had been in field operation for approximately 27 years. The investigated pipes had reached the end of their service life and were removed from operation. The PUR foam functions as the thermal insulation and structural support material between the steel service pipe and the outer casing of the district heating pipe system. Visual inspection of the recovered samples revealed a distinct color gradient within the foam structure (see Figure 1 Measurement locations on a PUR foam sample). The region of the foam directly adjacent to the steel service pipe exhibited strong darkening and appeared almost black in color. Moving away from the steel pipe toward the outer casing, the foam gradually became lighter, indicating a gradient in the degree of ageing or chemical modification across the insulation layer. Foam specimens were therefore collected from different positions within the insulation thickness in order to evaluate potential differences between the steel-side interface region and the foam regions closer to the casing.

FTIR Spectroscopy (ATR Mode)

FTIR measurements were performed using the Attenuated Total Reflection (ATR) technique in order to analyze chemical changes in the polyurethane structure caused by long-term ageing. Initial FTIR measurements were carried out directly on the darkened surface layer of the foam. Subsequently, approximately 1 mm of the surface layer was mechanically removed, and the measurement was repeated on the newly exposed material. This approach allowed the comparison between the surface layer and the underlying bulk material. The spectra were analyzed with particular focus on characteristic polyurethane absorption bands, including those associated with urethane linkages, carbonyl groups, and C–H stretching vibrations. The measurements were used to evaluate the presence or absence of identifiable polymer bond structures in both the surface and subsurface layers of the foam.

X-Ray Diffraction (XRD)

XRD analysis was conducted to investigate whether crystalline inorganic phases were present within the darkened surface layer of the aged PUR foam. The objective of this analysis was to determine whether materials originating from the steel service pipe, such as iron oxides, had migrated into the foam and could potentially influence the FTIR measurements. Samples from the surface region of the foam were analyzed using XRD to identify any crystalline phases. The diffraction patterns were evaluated and compared with reference patterns for common iron oxide phases.

Chemical Detection of Iron Compounds

To further investigate the possible presence of iron species within the foam, qualitative chemical analysis was performed using an acid extraction followed by colorimetric detection. Polyurethane foam samples were treated with nitric acid (HNO_3) to extract potential metal ions from the material matrix. The resulting solution was subsequently reacted with potassium ferrocyanide ($K_4[Fe(CN)_6]$), which forms a characteristic blue complex (Prussian blue) in the presence of iron species.

This procedure was applied to assess whether iron-containing materials originating from the steel service pipe had migrated into the foam structure during long-term operation.

Results

FTIR spectroscopy using the ATR technique was performed on the PUR foam samples recovered from district heating pipes that had been in service for approximately 27 years. The spectra obtained were analyzed to identify the characteristic functional groups associated with the polyurethane structure. Assignment of the absorption bands was carried out according to previously reported studies on PUR foams and their degradation behavior (Doyle und Weidlich 2021; França De Sá u. a. 2017; Hatchett, Kinyanjui, und Sapochak 2007). To ensure the reliability of the measurements, repeatability tests were performed, and the resulting spectra showed a high degree of overlap when measured repeatedly at the same location, confirming the consistency of the FTIR measurements. The analysis was conducted at four different positions across the insulation thickness in order to investigate possible spatial variations in chemical structure associated with long-term ageing.

The investigated locations included:

1. The interface between the PUR foam and the steel service pipe
2. A depth of approximately 1 mm from the steel interface toward the casing
3. A depth of approximately 5 mm toward the casing
4. The interface between the PUR foam and the outer casing

The measurement locations are illustrated in Figure 1 Measurement locations on a PUR foam sample.

Figure 1 Measurement locations on a PUR foam sample

The FTIR spectra revealed a pronounced difference between the foam region directly adjacent to the steel service pipe and the regions located further inside the insulation layer. Figure 2 FT-IR spectrum of naturally aged polyurethane foam insulation from a district heating pipeline after 27 years of in-field operation. illustrates the spectrum measured at the immediate interface shows a substantial deviation from the spectra obtained in the interior regions of the foam. At the steel–foam interface, where the material visually exhibited strong darkening and appeared nearly black, most of the characteristic absorption bands associated with the polyurethane structure were significantly reduced or nearly absent. In particular, absorption bands related to key functional groups of PUR, including isocyanurate structures, urethane carbonyl groups (C=O), ether linkages (C–O–C), and N–H stretching vibrations, were either strongly diminished or completely lost in the spectrum. The mentioned functional groups have been used in multiple studies for identification and degradation purposes (França De Sá u. a. 2017; Hatchett, Kinyanjui, und Sapochak 2007; A. Vega, Yarahmadi, und Jakubowicz 2018). In contrast, the FTIR spectra obtained from the 1 mm and 5 mm depths toward the casing exhibited clear and well-defined absorption bands corresponding to these functional groups. The spectra recorded at these locations showed a high degree of overlap with each other, indicating that the underlying foam material retained a consistent molecular structure across these regions (Jakubowicz u. a. 2022). The reappearance of the characteristic PUR bands after removal of approximately 1 mm of the surface layer suggests that the spectroscopic signal disruption is confined primarily to the outermost region of the foam adjacent to the steel pipe. The spectrum obtained at the foam–casing interface also displayed identifiable polyurethane absorption bands, although slight variations in intensity were observed compared with the spectra obtained at intermediate depths. The observed disappearance of characteristic PUR absorption bands at the steel interface represents a significant challenge when using FTIR spectroscopy to assess the degradation state of long-term field-aged district heating insulation materials. In cases where the foam surface has undergone severe discoloration, resulting in a dark or nearly black appearance, the altered surface layer can interfere with infrared measurements and obscure the characteristic absorption features typically used to evaluate polymer degradation. This phenomenon complicates the establishment of reliable spectroscopic benchmarks for determining the end-of-service-life condition of PUR insulation in district heating pipes. When FTIR measurements are performed directly on such darkened surfaces, the absence of identifiable absorption bands could potentially lead to incorrect conclusions regarding the chemical state of the material.

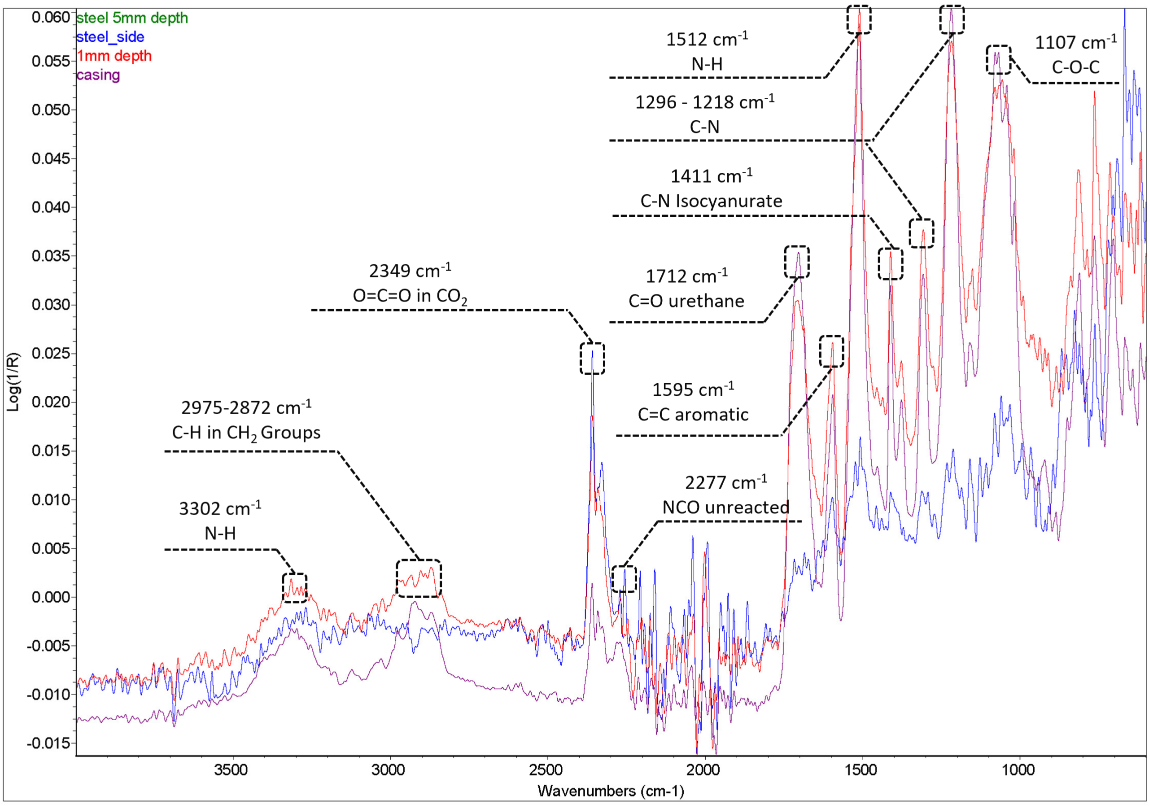

Figure 2 FT-IR spectrum of naturally aged polyurethane foam insulation from a district heating pipeline after 27 years of in-field operation.

The loss of spectral features, especially within the range of 2000 to 1000 cm^{-1} at the steel interface therefore raises an important question regarding the underlying cause of the signal disruption. One possible explanation could be the presence of inorganic contaminants, such as iron ions or iron oxide particles originating from corrosion processes in the steel service pipe. Such species could potentially interfere with infrared absorption or modify the surface characteristics of the foam.

To investigate whether inorganic phases were present in the darkened foam region located at the interface with the steel service pipe, XRD analysis was performed. The purpose of this analysis was to determine whether crystalline corrosion products, particularly iron oxides originating from the steel pipe, had migrated into the polyurethane foam during long-term operation and could potentially influence the FTIR measurements. XRD is commonly used for the identification of crystalline phases in materials and is widely applied in corrosion studies to detect iron oxide products formed during steel oxidation processes (Cornell und Schwertmann 2003; Cullity und Stock 2001). Samples taken from the darkened interface region of the foam were subjected to XRD analysis, and the obtained diffraction patterns were compared with reference patterns of common iron oxide phases, including hematite (Fe_2O_3), magnetite (Fe_3O_4), and goethite (FeOOH). These phases represent the most frequently observed corrosion products formed during the oxidation and environmental degradation of iron and steel (Cornell und Schwertmann 2003; Revie und Uhlig 2008). Under long-term service conditions, corrosion products from steel components may in some cases migrate into adjacent materials or accumulate at interfaces, potentially altering local chemical environments (Revie und Uhlig 2008). Figure 3 Diffractometer Analysis of Foam–Pipe Interface illustrates that the XRD patterns obtained from the investigated foam samples within the 10–80° (2θ) scan range do not show any detectable diffraction peaks corresponding to crystalline iron oxide phases. Instead, the diffraction pattern exhibits a broad amorphous halo, which is characteristic of polymeric materials such as polyurethane. The sharp diffraction peaks expected for common iron oxide phases are indicated in red in the figure; however, no such reflections are observed in the measured pattern. Polymers typically lack long-range crystalline order and therefore produce diffuse diffraction patterns rather than sharp Bragg peaks in XRD measurements (Alexander 1985; Sperling 2006). The absence of identifiable crystalline peaks suggests that no significant amount of crystalline inorganic phases is present within the analyzed foam region.

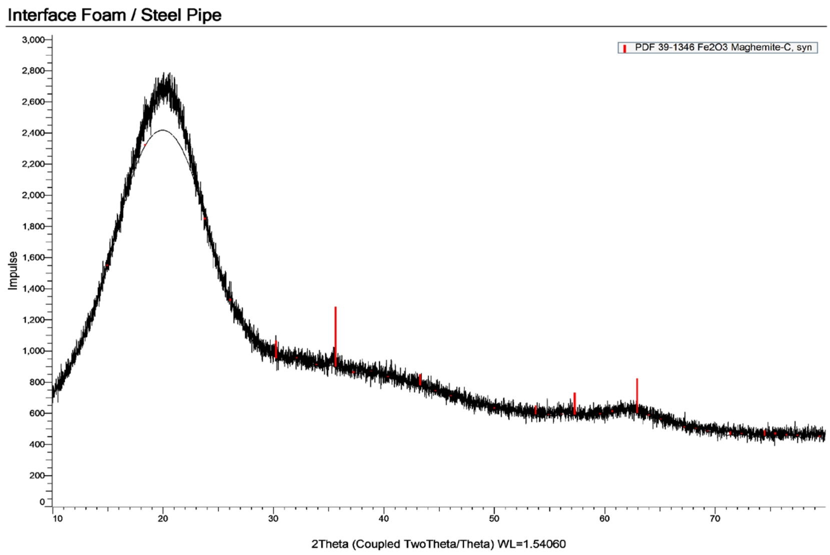

Figure 3 Diffractometer Analysis of Foam–Pipe Interface

These findings indicate that the darkened surface layer observed at the steel–foam interface is not associated with the presence of crystalline corrosion products from the steel service pipe. Consequently, the disappearance of FTIR absorption bands at this interface cannot be attributed to the presence of crystalline iron oxide phases within the foam structure.

In order to further evaluate the possibility of iron ion migration from the steel service pipe into the polyurethane foam, qualitative chemical analyses were performed to detect the presence of ferric ions (Fe^{3+}). The detection method was based on the well-known reaction between iron(III) ions and potassium ferrocyanide ($K_4[Fe(CN)_6]$), which results in the formation of a deep blue complex commonly referred to as Prussian blue. This reaction is highly sensitive and widely used for the qualitative identification of ferric ions (Harris 2007; Skoog u. a. 2022). The formation of Prussian blue results from the coordination reaction between ferric ions and hexacyanoferrate complexes, producing the characteristic intense blue precipitate that allows visual detection of Fe^{3+} ions even at relatively low concentrations (Inorganic Chemistry 2018). Two different approaches were applied during the chemical analysis. First, the darkened foam material taken from the interface region was directly exposed to a solution containing potassium ferrocyanide ($K_4[Fe(CN)_6]\cdot 3H_2O$) in order to observe whether a color reaction indicating the presence of Fe^{3+} ions would occur. Second, foam samples were subjected to acid digestion using nitric acid (HNO_3) to extract any potential metal ions embedded within the polymer matrix. Acid digestion is commonly used in analytical chemistry to dissolve solid samples and release metal ions into solution prior to qualitative or quantitative analysis (Harris 2007; Skoog u. a. 2022). The resulting solution was then reacted with potassium ferrocyanide to test for the presence of dissolved ferric ions.

In both cases, no characteristic blue coloration associated with Prussian blue formation was observed. This result indicates that detectable concentrations of ferric ions were not present in the analyzed foam samples (see Figure 4 Chemical Analysis for Detection of Iron Oxides at the Pipe–Foam Interface in PU Insulation). The chemical tests therefore support the XRD findings, suggesting that iron ions or corrosion products originating from the steel pipe have not significantly migrated into the polyurethane foam. The absence of detectable iron species further suggests that the observed loss of FTIR absorption signals at the interface region cannot be attributed to interference caused by metallic contamination or corrosion products.

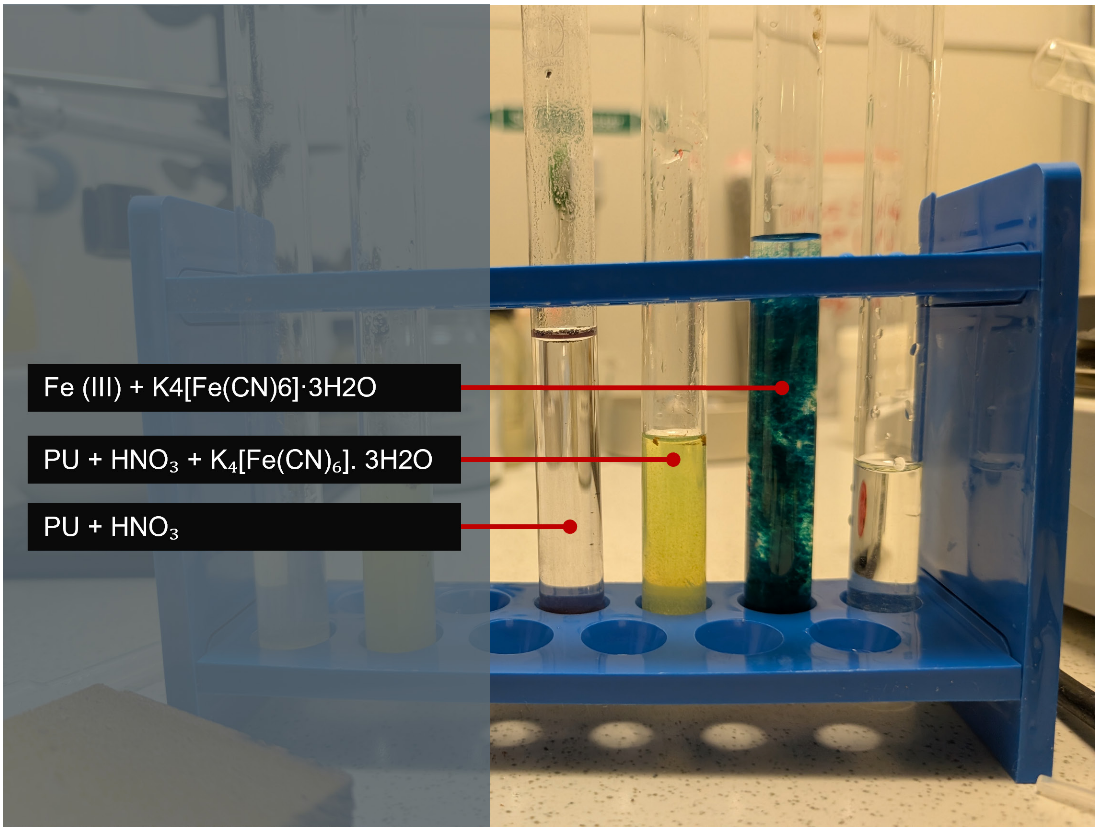

Figure 4 Chemical Analysis for Detection of Iron Oxides at the Pipe–Foam Interface in PU Insulation

To further investigate possible structural changes within the polyurethane foam resulting from long-term service conditions, nuclear magnetic resonance (NMR) analysis was performed with particular focus on hydrogen distribution within the material. NMR spectroscopy is widely used for the characterization of polymer structures because it provides detailed information about the local chemical environment and mobility of hydrogen atoms within polymer chains (Claridge 2016; Keeler 2011). In polymer systems, variations in NMR signals can reveal changes in molecular structure, cross-linking density, and degradation processes that occur during ageing (Mehring 1996; Schmidt-Rohr und Spiess 1994). The NMR measurements were conducted across the insulation thickness in order to examine potential variations in hydrogen-related signals between the steel pipe interface and the outer casing region. This approach allowed the evaluation of possible gradients in polymer structure that may have developed during the extended operational lifetime of the pipe. Spatial variations in NMR signals have previously been used to investigate structural heterogeneity and ageing gradients in polymeric materials exposed to long-term environmental or thermal stress (Schmidt-Rohr & Spiess, 1994; Claridge, 2016). The results indicated variations in the hydrogen-related signals across the insulation layer, suggesting changes in the molecular environment of hydrogen atoms within the polymer structure. These variations were more pronounced in the region adjacent to the steel service pipe, where the foam exhibited the strongest visual discoloration and where FTIR measurements showed the most significant loss of characteristic absorption bands (see Figure 5 . Change in hydrogen signal intensity as a function of depth, measured by NMR.). Changes in NMR signals in polymer systems are often associated with modifications in chain mobility, chemical bonding, or cross-linking within the polymer matrix (Keeler 2011; Schmidt-Rohr und Spiess 1994). The observed changes in the NMR signals therefore suggest that structural modifications within the polyurethane matrix may have occurred in the interfacial region during long-term operation, potentially associated with thermal exposure or other ageing processes. Thermo-oxidative degradation of polyurethane materials can lead to chain scission, cross-linking reactions, and the formation of new chemical structures, which may alter the local hydrogen environments detectable by NMR spectroscopy (Doyle und Weidlich 2021; França De Sá u. a. 2017). Such changes may contribute to the formation of the darkened surface layer observed at the steel interface, where the ageing conditions are expected to be most severe.

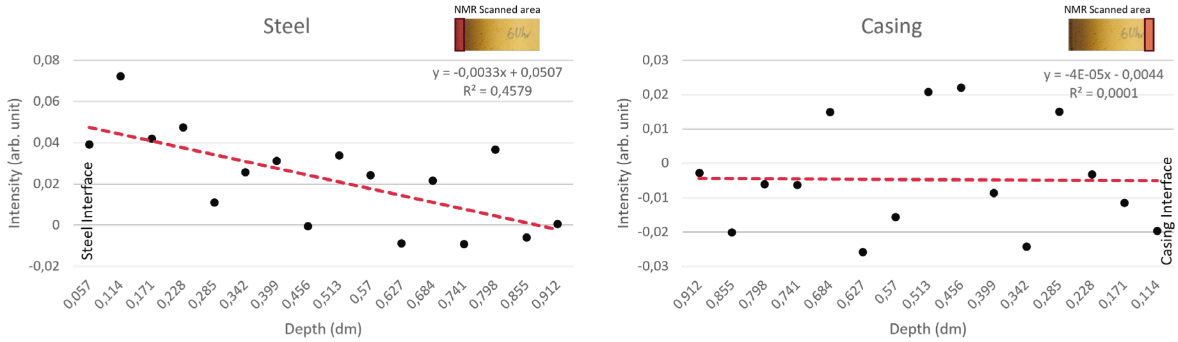

Figure 5 . Change in hydrogen signal intensity as a function of depth, measured by NMR.

Taken together, the results from FTIR, XRD, chemical analysis, and NMR indicate that the loss of FTIR absorption bands at the steel–foam interface is not related to the presence of iron ions or crystalline corrosion products. Instead, the findings suggest that the phenomenon is likely associated with ageing-related modifications of the polymer structure occurring in the interfacial region during long-term service.

Discussion

The FTIR analysis revealed that the PUR foam located directly at the interface with the steel service pipe exhibits a strong darkening, accompanied by the disappearance of most characteristic infrared absorption bands associated with polyurethane functional groups. In contrast, spectra obtained from locations only 1 mm beneath the interface display well-defined absorption bands corresponding to urethane carbonyl groups (C=O), ether linkages (C–O–C), N–H stretching vibrations, and isocyanurate structures. These functional groups are typically used as diagnostic absorption bands in FTIR analysis of polyurethane materials (França De Sá u. a. 2017; Hatchett, Kinyanjui, und Sapochak 2007). The observation therefore suggests that the loss of FTIR spectral features is limited to a very thin surface layer and is not representative of the bulk polymer structure. One plausible explanation for the disappearance of FTIR signals is the formation of a highly altered surface layer resulting from long-term thermo-oxidative degradation of the polyurethane matrix. Prolonged exposure to elevated temperatures and oxidative conditions can lead to extensive chemical transformations within the polymer network, including chain scission, cross-linking reactions, and the formation of new oxidation products (Doyle und Weidlich 2021; França De Sá u. a. 2017). During advanced degradation stages, these reactions may lead to the formation of conjugated carbon-rich structures within the polymer matrix, which are commonly associated with progressive discoloration and darkening of polymeric materials (Grassie und Scott 1988).

Such carbon-rich or highly conjugated materials typically exhibit broad and featureless infrared absorption behavior due to their disordered molecular structure and extensive electronic delocalization. In addition, darkened or partially carbonized surfaces tend to strongly absorb and scatter infrared radiation. As a result, the incident infrared beam used in ATR-FTIR measurements may be significantly attenuated before it can interact effectively with the underlying polymer bonds responsible for the characteristic absorption peaks. Consequently, the FTIR spectrum obtained from this altered surface layer may appear largely featureless or show a substantial reduction in the intensity of the expected polymer absorption bands (Stuart 2008). Another contributing factor may be the optical properties of the blackened surface layer. Dark materials generally exhibit high broadband absorbance across the infrared spectral range, which can reduce the penetration depth of the evanescent infrared wave generated during ATR measurements. Because ATR-FTIR typically probes only the upper few micrometers of a material surface, the presence of an optically dense or highly absorbing layer can effectively prevent the spectroscopic detection of the underlying polymer structure (Griffiths und De Haseth 2007; Stuart 2008).

The results of the XRD and chemical analyses further support this interpretation. The absence of detectable iron oxide phases and the lack of ferric ion detection indicate that the signal suppression cannot be attributed to inorganic contamination originating from the steel service pipe. Instead, the phenomenon appears to be associated primarily with intrinsic chemical modifications of the polyurethane surface resulting from long-term ageing.

PUR foam in district heating pipe assemblies performs several essential functions that directly influence the service life of the system. In addition to acting as thermal insulation, the foam contributes to the structural integrity of the pipe assembly by supporting the steel service pipe, maintaining the bond between the steel pipe and the outer casing, and restricting pipe movement caused by thermal expansion (Alberto Vega, Yarahmadi, und Jakubowicz 2021). During long-term operation, however, PUR foam is exposed to thermal, chemical, and environmental stresses, which gradually lead to degradation of the polymer network and a loss of its initial mechanical and thermal performance (Doyle und Weidlich 2021; França De Sá u. a. 2017). Consequently, reliable analytical methods are required to evaluate the ageing state of the insulation material and to support lifetime assessment of district heating pipelines.

Conclusion

FTIR is widely used to study degradation mechanisms in polyurethane materials because it enables the identification of changes in molecular bonds within the polymer structure. In the present investigation, PUR foam samples taken from district heating pipes that had reached the end of their service life were analyzed using FTIR and complementary characterization techniques. The outer surfaces of the recovered foams were strongly darkened, and FTIR measurements performed directly on these surfaces showed a substantial reduction or complete loss of characteristic absorption bands associated with polyurethane structures. However, when approximately 1 mm of the surface layer was mechanically removed, the typical FTIR spectral features reappeared, indicating that the underlying material still retained detectable molecular characteristics. This observation suggests that the surface layer formed during long-term service interferes with infrared measurements and may mask the spectral information of the underlying polymer.

To determine whether this effect could be related to contamination from the steel service pipe, XRD and chemical analyses were performed to identify possible iron oxide phases or metallic ion migration into the foam. The results showed no detectable traces of iron oxides or other metallic compounds on the foam surface. In addition, NMR analysis was conducted to investigate potential changes in hydrogen distribution across the foam structure, particularly from the steel pipe interface toward the outer casing.

The combined results indicate that the loss of FTIR absorption bands at the foam surface is not associated with metallic contamination but rather with surface modifications that develop during long-term ageing in service. According to (França De Sá u. a. 2017; Grassie und Scott 1988; Stuart 2008), it is hypothesized that these modifications are associated with advanced thermo-oxidative degradation of the polyurethane, leading to the formation of carbon-rich structures, which are known to accompany discoloration in aged polymers. Given the surface sensitivity of ATR-FTIR measurements, such a highly absorbing surface layer may attenuate the infrared signal and obscure the characteristic absorption bands of the underlying materia (Stuart 2008). These findings highlight an important limitation of FTIR when applied directly to severely aged PUR insulation materials. Surface conditions can significantly influence spectroscopic measurements and may lead to misinterpretation of degradation levels if sample preparation is not carefully considered. Therefore, removing the altered surface layer and employing complementary analytical techniques are recommended when assessing the ageing state of PUR foam in district heating systems.

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