Protocols for testing materials in contact with disinfected water, integrating batch- and continuous-flow conditions

Deliverable D1.5, WP1

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Abstract

This deliverable focuses on assessing the migration behavior of various materials such as pipes, seals and coatings used in drinking water distribution networks. Several types of plastics, epoxy resins, cement mortars and antioxidants were tested to determine their impact on water quality and disinfectant stability. Two chemical disinfectants were used for generalisation purposes, primarily sodium hypochlorite (NaOCl) and, for comparison, chlorine dioxide (ClO_2). A new comprehensive methodology was used for this study. The materials were ground to powder using a CyroMill that was constantly cooled with liquid nitrogen. These powders were tested for maximum potential release of organic compounds, consumption of disinfectant and formation of disinfection by-products (DBPs). Comparative migration studies with powder materials were also carried out with standardized methods for the approval of materials in contact with drinking water. In addition, experiments were carried out in a pipe loop to assess the role of the contact mode, considering that standardized methods rely on batch tests.

The results indicate that while plastic pipes contribute less to the formation of DBPs, rubber seals and epoxy resins may have a significant impact on water quality. Also cement mortars have an impact mainly due to the organic additives present in the cement formulations. The research outcomes provide a comprehensive evaluation of current testing protocols and suggest potential improvements for the management of materials in drinking water systems.

The new powder tests showed higher levels of DOC and DBPs in water for most migrations compared to the conventional pipe tests. In almost all cases, a higher level of release was observed when disinfecting with ClO_2, which is more reactive than NaOCl. The adsorption of DBPs on powders and other involved reactions result in a complicate interaction between the different compartments. The topic could not be clarified in detail, so further research is suggested.

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Table of contents

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Abbreviations

AI	Aggressivity Index
AO	Antioxidant
BDCM	Bromodichloromethane
D	Deliverable
DBPs	Disinfection by-products
DCAN	Dichloroacetonitrile
DOC	Dissolved Organic Carbon
DOM	Dissolved Organic Matter
DPD	N,N-diethyl-1,4-phenylenediamine
DW	Drinking Water
DWDN	Drinking Water Distribution Network
DWTP	Drinking Water Treatment Plant
EC	European Commission
EDTA	Ethylenediaminetetraacetic Acid
EPDM	Ethylene Propylene Diene Monomer Rubber
GA	Grant Agreement
GC	Gas Chromatography
HAAs	Haloacetic Acids
HDPE	High Density Polyethylen
HPLC	High Performance Liquid Chromatography
LC	Liquid Chromatography
MS	Mass Spectrometry
MP	Microplastics
NBR	Nitrile-Butadiene Rubber
NOM	Natural Organic Matter
OM	Organic Matter
PA	Polyamide
PB	Polybutene
PE	Polyethylene
PE-RT	Polyethylene Raised Temperature
PE-X	Cross-linked Polyethylene
PE-Xa	Peroxide Cross-linked Polyethylene
PE-Xb	Silane Cross-linked Polyethylene
PE-Xc	Irradiation Cross-linked Polyethylene
POM	Polyoxymethylene
PTFE	Polytetrafluoroethylene
S/V	Surface Area To Volume Ratio
SAC	Spectral Absorption Coefficient
TCM	Trichloromethane
TIC	Total Inorganic Carbon
THM	Trihalomethane
TOC	Total Organic Carbon
UV	Ultraviolet
VF	Vulcanized Fibre
WP	Work Package

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Introduction

Plastic pipes are common materials used in the extraction, treatment and distribution of drinking water. Epoxy resins are widely used for relining of old and damaged pipes in drinking water distribution networks (DWDN). The majority of drinking water is treated at a central facility and subsequently transported to individual house connections via the DWDN. Inside the buildings, the water is piped to the extraction points via the drinking water installation, where it can flow out as needed via suitable taps.

After extraction, during treatment and distribution, water comes into contact with a wide range of materials and substances. In drinking water treatment plants (DWTP) and DWDN, migration of pipes materials constituents into drinking water disinfectants might promote the release and formation of substances. In drinking water installations in buildings, high surface-to-volume ratios, elevated temperatures and longer residence times can favour the migration of substance-bound organic molecules into drinking water.

In this research project, selected materials such as pipes, pipe coatings, fittings and seals, which are considered to be the starting point for migrating substances, were examined with regard to reaction products of reactions with disinfectants. Batch and loop experiments were carried out to simulate specific conditions that may occur in real installations. For this purpose, the plastic materials were brought into contact with chlorinated and unchlorinated water under defined conditions and the resulting solutions were analysed for dissolved organic carbon (DOC), chlorine consumption, toxicity and disinfection by-products (DBPs).

Whereas in the past, metal pipes were predominantly used for drinking water installations, plastics are increasingly being used as pipework materials. The polymers used to manufacture plastic materials are characterised by very large, i.e. long-chain molecules, which can also be cross-linked to varying degrees. The main materials used for drinking water pipes are the following: cross-linked polyethylene (PE-X), which is achieved mainly by the use of a peroxide addition (PE-Xa), the silane method (PE-Xb), or irradiation (PE-Xc), polybutene (PB), polyethylene with increased heat resistance (PE-RT) and polypropylene (PP). In pipework for cold water additionally pipes made of polyvinyl chloride (PVC) and polyethylene (PE) are installed (Isaacson et al., 2021; Mohammadi et al., 2022; Zhang et al., 2023). Polyoxymethylene (POM), polyamide (PA), ethylene propylene diene monomer rubber (EPDM) and polytetrafluoroethylene (PTFE) are commonly used for fittings and seals (Zhang et al., 2023).

The polymers consist of compounds that are not water soluble due to the size of the molecules (Dopico-García et al., 2003). However, in addition to the polymer, the plastics might also contain residues of unreacted monomers, resins and oligomers. Different types of compounds may be leached depending on the production process and the manufacturer. Some plastic polymers, such as PE-X, are additionally cross-linked to achieve increased temperature resistance, durability and corrosion resistance (Baur, 2013; Brunner, 2014). The substances added for this purpose, such as organic peroxides and their reaction products, can also migrate into the drinking water (Brocca et al., 2002; Lund et al., 2011). In addition, additives are used that have a function in the manufacture or use of the plastics. These include stabilisers, which are designed to protect the plastic from oxidative attack. Polymerisation aids are added to initiate or control polymerisation. These may be metal compounds that remain in the finished product and may therefore leach into the drinking water. In addition, fillers, pigments, flame retardants, accelerants, lubricants and solvents may be present in the plastic (Hiles et al., 2019; Jemec Kokalj et al., 2022; Samarth and Mahanwar, 2021). In contrast to polymers, stabilisers (primarily antioxidants (AO)) or their degradation and conversion products have a much smaller

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molecular size and potentially greater mobility. For this reason, these organic substances can enter drinking water and thus also affect its quality (Whelton and Nguyen, 2013).

The manufacturers do not disclose in detail which substances are used to produce the individual products. The recipe is only disclosed as part of the product certification. However, this information is not available when analysing products on the market or in real drinking water installations.

Several studies have been conducted on compounds that can be released from plastic pipes (Diera et al., 2023; Pelto-Huikko et al., 2021; Ryssel et al., 2015). Additionally studies focused on the effects on DW odour and taste (Durand and Dietrich, 2007; Kalweit et al., 2019; Lund et al., 2011) . Only a few studies have focused on the interactions between materials that come into contact with drinking water and chlorine, investigating disinfectant consumption (Cao et al., 2020), contaminant leaching and formation of DBPs.

Furthermore, to date, a risk assessment approach investigating the potential increase in NOM due to the contact of pipes with water have rarely been applied (Phillips et al., 2021). All these aspects make it difficult to plan potential disinfectant strategies, to select materials according to their properties and composition and to support policy makers in the selection of appropriate materials for contact with disinfected DW. Furthermore, literature studies have not usually used comprehensive approaches to investigate the interaction between water quality, pipe material and disinfectant.

For this study, the interactions of ten plastic pipes, one antioxidant commonly used in their manufacture (Irganox 1010®), two fittings, three epoxy resins and one cement mortar lined pipe with and without chemical disinfectants (NaOCl, ClO_2) were examined.

Studies (Diera et al., 2023; Zhang et al., 2023) have shown that plastic pipes and their antioxidants and degradation products contribute a relatively small percentage of the total amount of compound measured in the DWDN. Instead, a larger proportion of the detected compounds was attributed to rubber seals used in the water system, which is why four seals were also included in this study.

The powder test method is introduced as a new test protocol and is compared with the common standardised method (DIN EN 12873-1). In addition, continuous experiments were carried out in a loop with tubes. To get an overview of the organic matter content in the migration waters, the dissolved organic carbon (DOC) as a sum parameter is determined. However, this parameter does not provide any information about the identities of the substances. Therefore, further investigations were carried out to characterise the organic matter using size exclusion liquid chromatography coupled with continuous organic carbon detection (LC-OCD), absorbance spectra and fluorescence matrices.

The chlorine consumptions of the different materials were measured, together with conventional DBPs generated by NaOCl and ClO_2. All results were comprehensively evaluated to assess knowledge gaps and suggest further research.

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Materials and Methods

Pipes, Fittings and Seals

All used pipes were purchased on the local market during the last 24 months and are listed in Table 1. All tested pipes are approved for contact with drinking water and have inner diameters between 12 and 16 mm and thicknesses between 1.9 and 2.1 mm.

Table 1: Overview of the tested materials with their types, manufacturers/origins and surface-to-volume ratios

Type	Material	Manufacturer	Country	S/V [dm^{-1}]
Pipe	PE-Xa	Rehau	Switzerland	34
Pipe	PE-$Xb_{[s]}$	Tiemme	Italy	33
Pipe	PE-$Xb_{[b]}$	Tiemme	Ítaly	25
Pipe	PE-Xc	Becker Plastic	Germany	31
Pipe	PA	Pipelife	Austria	34
Pipe	PE 80	Kirchhoff	Germany	32
Pipe	PE 100 RC	ADUXA	Germany	30
Pipe	PE RT	Becker Plastic	Germany	31
Pipe	PP	Aqua kessel	Germany	33
Pipe	PVC - U	Rehau	Switzerland	26
Fitting	PA	Tecuro	Germany	
Fitting	POM	Pipelife	Austria	
Seal	EPDM	Köro	Netherlands	
Seal	Aramid	Köro	Netherlands	
Seal	Vulcanised fibre	Köro	Netherlands	
Seal Tape	PTFE	Fermit	Germany	

Epoxy resins

Three epoxy resins approved for contact with drinking water were tested. They are used primarily in the so-called relining process. This involves renovating old water pipes by lining them with epoxy resin, which is a cost-effective alternative to replacing the pipes. A two-component epoxy resin from RS Technik AG (Switzerland)(subsequently referred to as resin 1) was tested, along with a resin from Norditube (Italy)(resin 2) and a resin from In.Tec (Italy)(resin 3).

Cement Mortar

A cast iron pipe with an outer diameter of 100 mm lined with cement mortar was supplied by Acque Bresciane S.r.l (Italy), one of the water supply companies that manages the drinking water supply in the province of Brescia. The cast iron pipes comply with EU Directive GB/33/EEC. The cement mortar lining consists of CEM III cement which is composed of Portland clinker and blast furnace and complies with the requirements of DIN EN 197-1 (2011).

Antioxidant

Irganox 1010® (Sigma Aldrich, USA) (3,5-di-*tert*-butyl-4-hydroxyhydrocinnamat), a commonly used antioxidant in plastic pipes production (Coron, 2008), was tested in powder form.

Chemicals

All chemicals were used as received without any further purification steps. Sodium hypochlorite solution (NaOCl) with 6-14% active chorine was purchased from Merck (Darmstadt, Germany). In the same week as each experiment, a 1.9 g/L ClO_2 stock solution (that corresponds to 5 g/L Cl_2) was prepared by adding two reagents in tablet form, TwinOxide® (Best, Netherlands), in 100 mL of

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ultrapure water. The components included sodium chlorite and additives and sodium bisulfate monohydrate and additives. All stock solutions were stored in headspace-free amber glass vials at 4 °C. Ultrapure water was obtained from a Merck Milli-Q IQ 7000 Ultrapure Water Purification System (Darmstadt, Germany).

Pre-treatment and test procedures

Before the test, adhesive spots, labels or inscriptions were removed from surfaces. By checking the test pieces in advance, it was ensured that the transport and storage conditions had as little influence as possible on the test results. The parts were stored at room temperature in a dark place and sealed with caps or aluminum foil.

Each test piece undergoes three phases according to DIN EN 12873-1 (2014) and DIN EN 1420 (2016):

(i) Rinsing: where possible, the test pieces were rinsed with running tap water for 60 ± 5 min at a speed of approximately 1 m/min. The test specimens that could not be rinsed were placed in a suitable vessel and rinsed for 60 ± 5 minutes with a constant flow of water from bottom to top at the same flow rate.

(ii) Stagnation with test water: the test pieces were placed in or filled with test water for a period of 24 ± 1 h at 23 ± 2 °C. The water was then drained.

(iii) Pre-wash: during pre-washing, the test pieces were rinsed again in the same way as in the first step (i). The test pieces were then washed with the test water for 2 minutes.

The migration test was carried out with a migration period consisting of 72 h contact time at 23 ± 2 °C for products approved to be brought into contact with cold drinking water. The test items were completely filled or immersed in the test water for the specified time, leaving no non-wetted surface. The tubes were sealed with glass plugs.

Current standard for cement mortar and modified procedure

Cement mortar, when tested in tube form, was processed according to a procedure inspired the technical standard DIN EN 14944-3 (2008). This procedure is structured in two stages:

(i) Pre-conditioning: it is divided into five steps. The first three last 24 h, the fourth lasts 72 h and the last one takes 24 h more.
(ii) Migration: this stage is made of three steps that last 72 h. The standard procedure requires this test to be done without disinfectant and using NaOCl at 1 $mgCl_{2\text{-}eq}$/L.

Compared to DIN EN 14944-3, the adopted procedure for cement mortar lining migration assessment had these differences:

- In the fifth step of pre-conditioning, 50 $mgCl_{2\text{-}eq}$/L of NaOCl were added. Hence, before the migration stage, the pipe was flushed at least five times to completely remove the disinfectant.
- During the three migration steps, additional disinfectant (rechlorination) was added daily or more frequently in case of excessive decay.
- ClO_2 was used as a disinfectant in addition to NaOCl.
- Three types of water were tested in the migration step and are shown in Table 2. The aggressivity index (AI) value was calculated according to Larson and Skold (Larson and Skold, 1958) as followed:

$$AI = pH + log(A \cdot H)$$

where A is the total alkalinity of the water (mg $CaCO_3$/L) and H is the hardness (°F).

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- Another difference from the standard procedure is the addition of two extra steps after migration stage:
  - Ageing: it is made of 24 steps, each one lasting 72 h. It was performed with both NaOCl and ClO_2 at 1 mg $Cl_{2\text{-}eq}$/L with daily rechlorination.
  - Post-ageing migration: this stage involves only one 72 h step. It was performed with NaOCl and ClO_2 at 1 mg$Cl_{2\text{-}eq}$/L with daily rechlorination. Moreover, additional re-disinfection was performed in case of disinfectant decay.

Table 2 Summary of characteristics of different waters used in experiments with cement mortar.

Type of Water	Pre-Conditioning	Standard	Type 1	Type 2	Lab tap water
Purity level	II	I	I	I	-
AI	11	11	11	13	12.3
pH	7.4	7	7	8.3	7.4
Hardness [mg/L $CaCO_3$]	100	100	104	196	-
Alkalinity [mg/L $CaCO_3$]	122	122	107	254	-
HCO_3^- [mg/L]	244	149	130	300	318
CO_3^{2-} [mg/L]	-	-	-	5	-
Ca^{2+} [mg/L]	80	35	25	45	110
Cl^- [mg/L]	142	61	35	95	38
Mg^{2+} [mg/L]	-	3	10	20	37
NO_3^- [mg/L]	-	2	25	25	39
Na^+ [mg/L]	84	15	49	109	-
SO_4^{2-} [mg/L]	-	7	45	35	48
K^+ [mg/L]	-	-	10	10	1.7

Testing Materials in powdered form

All materials were cleaned before milling according to DIN EN 12873-1 (2014) and DIN EN 1420 (2016) and dried for 24h. The internal parts of the pipes and other components were cut and divided into pieces of 3-5 mm. 2.2 g of the plastic pieces was then grinded through a cryogenic ball milling (Retsch CryoMill, Germany) and a ball of stainless steel with a diameter of 25 mm. The volume of the jar is filled with approximately 1/3 part of each grinding ball, pieces of material and air. The grinding jar (50 mL) was continually cooled with liquid nitrogen before and during the grinding process. The cooling process was carried out in a sequence of three cycles. The first cycle consisted of (i) a pre-cooling step with a frequency of agitation of the grinding jar of 5 Hz and duration of 20 minutes, (ii) a grinding step (frequency 30 Hz, duration 5 min), and (iii) an intermediate cooling step (frequency 5 Hz, duration 5 min). The other two cycles consisted only of the grinding and the cooling steps. Further details are reported by Eitzen et al. (Eitzen, 2019).

After milling, the powders were collected into amber glass vials with PTFE cap to avoid VOC losses and cross-contaminations, and stored in dark at environmental temperature. The mill was also used to grind the cement sample without liquid nitrogen.

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The number of particles of the plastic powders was determined using a particle counter (PAMAS SVSS, Rutesheim, Germany) based on laser light extinction measurements. Stock suspensions of 500 mL with a concentration of 10 mg/L of the powder were prepared using ultra-pure water (ELGA, Celle, Germany). The sizes of the particles were divided into 32 classes ranging from 1 µm to 200 µm.

To stabilize particles, a non-ionic surfactant (NovaChem, Postnova) was added to the suspension. Each stock suspension was analysed in five repetitions, according to the procedure suggested by Eitzen et al. (Eitzen, 2020). The suspension was constantly stirred during the analysis. Between the batches of different particles, the device was rinsed with 5 runs of ultrapure water.

All samples were prepared with ultrapure water and tested with the following disinfectant concentrations NaOCl (1, 10 and 50 mgCL_2/L) and ClO_2 (0.4, 1, 19, 50 mgCl_2/L) and as blanks without disinfection. All migration tests with the resulting powder and ultra-pure water were agitated with overhead rotators with a rotational frequency of 1 sec^{-1}. Flask with a volume of 1 L were mixed with the GFL 3040 (Burgwedel, Germany), vials of the sizes 10-50 mL were mixed with the Heidolph REAX2 (Schwabach, Germany). After a defined contact time (1, 3 and 7 d), residual disinfectant concentrations (performed immediately), organic matter and DBPs were measured. Samples intended for DBPs measurements were quenched with sodium thiosulphate. All samples were filtered through a 25 mm membrane with a pore size of 0.45 µm (CHROMAFIL Xtra PTFE, Macherey-Nagel, Germany) and stored in the dark at 4°C before analyses.

Blank and filter blank samples were prepared accordingly to the migration water samples, to exclude external contamination. In addition, blank residual chlorine tests to account for disinfectant decay in water without any materials were performed adopting the same procedures used for migration tests.

To compare the methods, a similar surface area to volume ratio (S/V ratio) was chosen for both tests. This involves the ratio between the surface area of the test piece (S) to come into contact with the test water and the volume of test water (V) per decimeter, i.e. in dm^{-1} (from dm^2/dm^3 or dm^2/L).

Loop Tests

For the loop tests, 4 m of pipe material was pre-washed in accordance with the standards (DIN EN 12873-1 and DIN EN 1420). The pipes were then filled with the test water and closed with a brass ball valve to ensure that as little air as possible remained in the pipe. A peristaltic pump (Masterflex, Avantor, USA) was connected and a constant volume flow of 1.5 L/min was set. Pumping tubes made of silicone with a diameter of 6.4 mm were connected using push-in fittings made of brass with a POM release ring and nitrile-butadiene rubber (NBR) seal. The contact time was 3 days, in line with the migration water tests according to DIN EN 12873-1 and DIN EN 1420 and in powdered form.

Analytical procedures

pH was measured by a pH meter (pH 340i, WTW, Weilheim, Germany). Temperature was measured during the whole time of the migration tests (Testo datalogger 175T1, Lenzkirch, Germany). Absorbance measurements were done with a UV/Vis (USA) spectrophotometer (Lambda 25, Perkin Elmer, USA), using a quartz cuvette with 10 mm optical path. For the UV light absorption at 254 nm wavelength (UVA254) the filtered samples were directly pipetted into the quartz glass cuvette and the absorbance was measured at wavelengths of 254 nm, 436 nm and 550 nm, respectively. Ultrapure water was used for zero adjustment and as a reference.

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TOC/DOC

All measurements of dissolved organic carbon (DOC) were carried out with a TOC analyzer (Elementar vario TOC cube, Langenselbold, Germany). The method used is based on DIN EN 1484 calibrated for DOC contents below 10 mg/L. For the DOC measurements, 10 mL of the sample was first filtered through a membrane filter with a pore size 0.45 µm and afterwards pipetted into a glass vials and spiked with 80 µL of 10% HCl to expel the TIC. The resulting carbon dioxide is automatically blown out before each measurement. All measurements were carried out as 3-fold determinations.

TOC analysis for the tested cement mortar tubes was performed according to DIN EN 1484 (2019).

Fluorescence

Fluorescence analyses was conducted using a fluorescence photometer (FluoroMax-4, Horiba, USA) with excitation wavelengths ranging from 200 to 550 nm (5 nm intervals), emission wavelengths from 240 to 600 nm (2 nm intervals) and a scan speed of 4800 nm/min. A quartz cuvette with a 10 mm optical path length and all four transparent faces was utilised. Prior to each analysis, the cuvette was rinsed with ultrapure water and subsequently with the sample.

LC-OCD

In addition to DOC measurements, the dissolved OM was characterized by liquid chromatography (LC) coupled with organic carbon detection (OCD) and UV detection (UVD) (DOC-Labor Huber, Germany). As described by Huber et al. (Huber, 2011), it separates NOM into its different fractions based on size and polarity, thus providing an analysis of the composition. The addition of UVD enhances the capabilities of LC-OCD by allowing the quantification of specific organic compounds within the NOM matrix. The chromatograms separate the chromatographic DOC (CDOC) into different fractions, including humic colloids, polysaccharides, humic substances, building blocks (fragments of humic substances), low molecular weight compounds (including low molecular weight acids), amphiphilic substances and neutral compounds.

Residual Chlorine

The determination of $Cl_{2\text{-eq}}$ concentrations in the NaOCl and ClO_2 stock solutions was done according to the iodometric method (Bridgewater et al., 2017).
Free chlorine and chlorine dioxide in samples were measured using the DPD method (DIN 38408-5 and DIN EN ISO 7393-2) based on oxidation N,N-diethyl-1,4-phenylenediamine (DPD) by free chlorine and formation of a red colour. Free chlorine and chlorite do not react under these conditions. HOCl and chlorine dioxide are reduced according to the following two partial equations:

$$Cl_2 + 2\ e^- \rightarrow 2\ Cl^- \qquad \text{Equation 1}$$

$$ClO_2 + e^- \rightarrow ClO_2^- \qquad \text{Equation 2}$$

In the reduction of Cl_2, two electrons are transferred and in the reduction of ClO_2, one electron is transferred. DPD acts as an electron donor. The colour intensity of the oxidised DPD was measured with a spectrophotometer at a wavelength of 510 nm and is proportional to the concentration of chlorine or chlorine dioxide.

Buffer solution was prepared using 60.5 g/L Na_2HPO_4 x 12 H_2O, 46 g/L KH_2PO_4 and 0.8 g/L of ethylenediaminetetraacetic acid (EDTA) in ultrapure water. For the DPD solution, 0.2 g EDTA was added to 1.1 g DPD in a dry 1000 mL volumetric flask, mixed with approx. 80 mL ultrapure water and shaken until the powders were dissolved. Then 2 mL of 95-98% sulfuric acid was added and the flask was filled up to 1000 mL. The two solutions were wrapped in aluminium foil for protection from light and heat.

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A 5 cm quartz cuvette was used for the measurements. Before each measurement, the cuvette was rinsed three times with ultrapure water. Each 0.68 mL buffer and DPD solution were placed in the cuvette. The cuvette was then filled up with 15 mL of the sample and immediately measured in the photometer at 510 nm. The samples from the migration tests with powder were filtered before measurement and then pipetted into the cuvette. In preliminary tests, alternative methods were also tested in which the sample was first mixed with DPD and then filtered. The difference between the two approaches was within the range of the measurement inaccuracies of the triplicate determinations.

The influence of the outside temperature on ΔC_R was also analysed. The DIN EN 12873-1 specifies a temperature range of 23°C ± 2°C. Therefore, migration water tests with selected pipe materials and Irganox 1010 were carried out at 21°C and 25°C ± 0.5°C as end points of these temperature ranges. The contact time was 72h.

DBPs

THM

For all experiments the concentrations of THM was measured with a headspace gas chromatography and mass spectrometer (HS-GC-MS, Agilent, Santa Clara, USA). The GC (Agilent 7890A) is equipped with a 60 m column (HP-5ms Ultra Inert, 19091S-436UI, Agilent) with an inner diameter of 0.25 mm and 0.25 µm film thickness. The MS (Agilent 5975C) uses an electron beam for ionization while filtering the ions with a quadrupole. The headspace sampling was performed by system (Gerstel MPS) using 20 mL amber glass bottles. The agitator shakes the samples for 40 min at 60 °C. 1 mL of gas sample is injected with a split 1:10 at 210°C in a split/split-less injector. The GC is operated with a flow of 0.6 mL/min of helium. The temperature program starts at 40 °C holding for 5 min, rising to 60 °C at 4 °C/min, then to 190 °C at 10 °C/min. The final temperature is hold for another 5 min. The run time per sample was 28 min. The MS operates in single ion monitoring mode scanning for the m/z 82, 83, 84, 85, 93, 117, 119, 124, 127, 129, 161, 163, 171, 173 and 174, each with a dwell time of 80 ms. The retention times are shown in Table 3. Triplicates were measure according to the respective standard (DIN 38407-30, 2007).

Table 3 Retention time (RT) of trichloromethane (TCM), bromodichloromethane (BDCM), chlorodibromomethane (DBCM) and tribromomehtane (TBM)

Analyte	**RT [min]**
TCM	8.18
BDCM	10.92
DBCM	14.03
TBM	15.70

HAA

Eight haloacetic acids (HAA8) including trichloroacetic acid (TCAA), bromodichloroacetic acid (BDCAA), chlorodibromoacetic acid (CDBAA), dichloroacetic acid (DCAA), bromochloroacetic acid (BCAA), dibromo acetic acid (DBAA), monochloroacetic acid (MCAA), and monobromoacetic acid (MBAA) were measured by high-performance liquid chromatography (Agilent 1290 Infinity II) coupled with triple quadrupole mass spectrometry (HPLC-MS/MS) systemA column (Luna Omega Polar) with 100 mm length, 4.6 mm inner diameter, 3 µm particle size and 100 Å pore size was used. The chromatography is operated with ultrapure water with 0.1% acetic acid (solvent A) and pure methanol (solvent B) by a gradient listed in Table 4 while maintaining a column temperature of 40 °C.

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Table 4 Gradient for flushing of LC column

Time [min]	solvent A [%]	solvent B [%]	Flow [mL/min]
0	99	1	0.8
1	99	1	0.8
9	5	95	0.8
10	5	95	0.8
11	99	1	0.8
14	99	1	0.8

The connected mass spectrometer (QTRAP 6500, Sciex) uses an electron spray ionisation in negative mode (CUR 35, CAD High, IS -4500, TEM 600, GS1 50, GS2 50, EP -10). Table 5 shows the multiple reaction monitoring program (MRM) for all measured quantifiers and qualifiers at the expected retention times. For the calibration, standards with 0.5, 1, 5, 10, 20, 40, 60, 80 and 100 µg/L are measured. The limit of quantification is at 0.5 µg/L for all HAA expect for DCAA at 0.1 µg/L.

Table 5 MRM program for all measured quantifiers and qualifiers at the expected retention times

	Quantifier						Qualifier						
HAA	Q1	Q3	Dwell Time [msec]	DP [V]	CE [V]	CXP [V]	Q1	Q3	Dwell Time [msec]	DP [V]	CE [V]	CXP [V]	expected RT [min]
CAA	93	35	30	-9	-16	-5	95	37	30	-9	-16	-5	2.39
DCAA	127	83	30	-5	-14	-9	129	85	30	-8	-13	-9	2.44
TCAA	161	117	30	-10	-12	-5	163	119	30	-15	-12	-11	4.45
BAA	137	79	30	-10	-22	-7	173	81	30	-15	-18	-9	2.73
DBAA	215	79	30	-10	-36	-9	217	81	30	-20	-34	-9	3.07
TBAA*	251	79	30	-30	-28	-9	253	81	30	-30	-28	-7	5.06
BCAA	171	79	30	-40	-28	-9	173	81	30	-35	-30	-9	3.07
BDCAA*	161	79	30	-5	-18	-7	163	81	30	-15	-18	-9	4.64

Sulfonic DBPs

Selected migration water samples with powder from pipe materials (PA, PVC, EPDM, PE-Xb) were analysed for sulfonic DBPs using supercritical fluid chromatography mass spectrometry (SFC-MS). The procedure is described in detail in the deliverable D1.1. Sodium thiosulfate (Merck, Germany) was used as quenching agent.

This project has received funding from the European Union's Horizon Europe research and innovation programme under grant agreement No 101081980.

Bioanalysis of migration water samples on a tailored panel of CALUX bioassays

Selected migration water samples with powder from pipe materials (PA, PE-Xc, PE-Xb) were analysed for bioactivity on a panel of CALUX assays. The procedure is described in the deliverable D1.3. Ascorbic acid (Merck, Germany) was used as the quenching agent for these samples.

Data management

Data of measurements in migration water tests for powders and pipes are deposited at the Zenodo SafeCREW community under DOI: 10.5281/zenodo.14012190.

Results

Particle size analysis

The sizes of the particles were divided into 32 classes ranging from 1 µm to 200 µm. The distribution of the particle sizes of all the materials is shown in Figure 1.

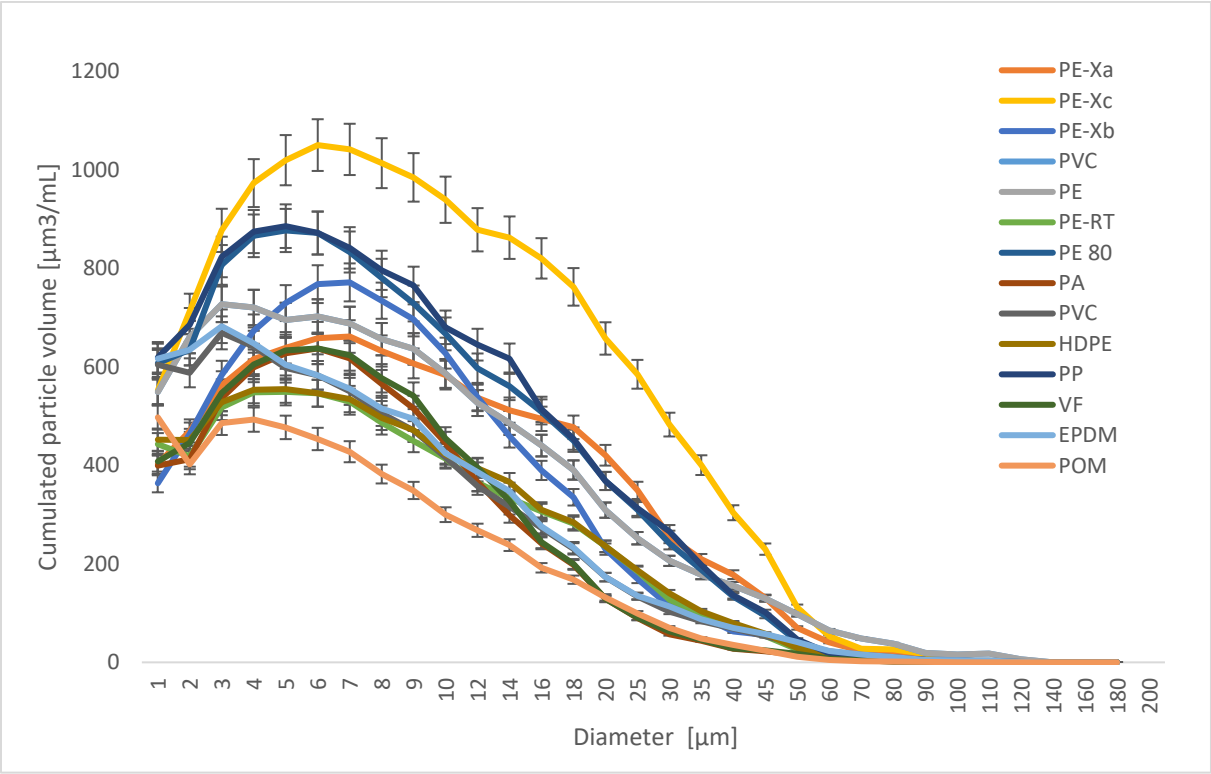

Figure 1 Particle volume distribution derived from a particle count in the diameter size classes for the cryo-milled plastic samples.

The particle size distributions for the different materials shown in Figure 1 were in similar ranges. The majority of the cumulated volumes of particle diameter classes were in the size range between 5 and 50 µm.

The particle distribution forms the basis for approximating the S/V ratio in the particle suspensions for the migration water tests. An S/V of 33 dm^{-1} was assumed as a comparative value, which corresponds to the average for all pipes not examined. To compare particles with pipes, the simplification was made that all particles have the shape of a sphere. Thus, the specific volume of the powder could be calculated, which resulted in an amount of 700 mg of material per litre of test water at the given S/V ratio.

This project has received funding from the European Union's Horizon Europe research and innovation programme under grant agreement No 101081980.

Disinfectant decay

For the determination of the chlorine consumption, a solution of NaOCl with 1 $mgCl_2/L$ was used. The residual chlorine content of the migration water of the samples with powered materials after 72 h can be seen in Figure 2.

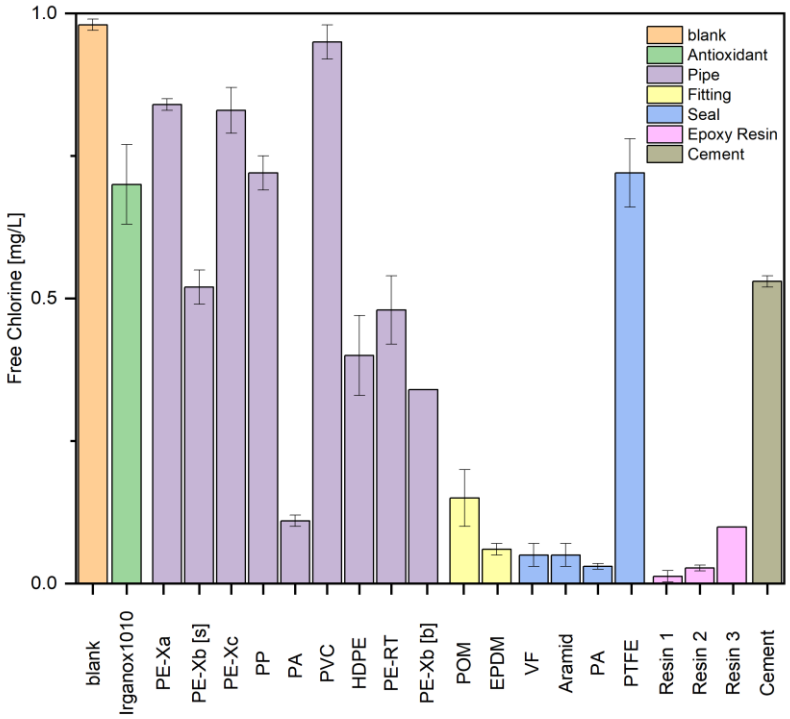

Figure 2 Free chlorine in migration water with powdered materials after 72h. Initial Chlorine concentration was 1 mg/L.

The observed disinfectant consumption is assumed to be related with the release of organic matter from the tested materials into the water and with the reaction of the disinfectant directly with the solid phase. The chlorine demand was highest for materials from the group of epoxy resins and sealing materials (with the exception of PTFE). For all migration water samples in which less than half of the original chlorine content was measured in the migration water after 72 hours, additional experiments with measurements after 15 and 60 min were carried out in order to obtain a time estimate of the chlorine demand as shown in Figure 33. The consumption in the presence of sealing material is faster than for epoxy resins and pipes. After 15 min, the chlorine was already completely consumed.

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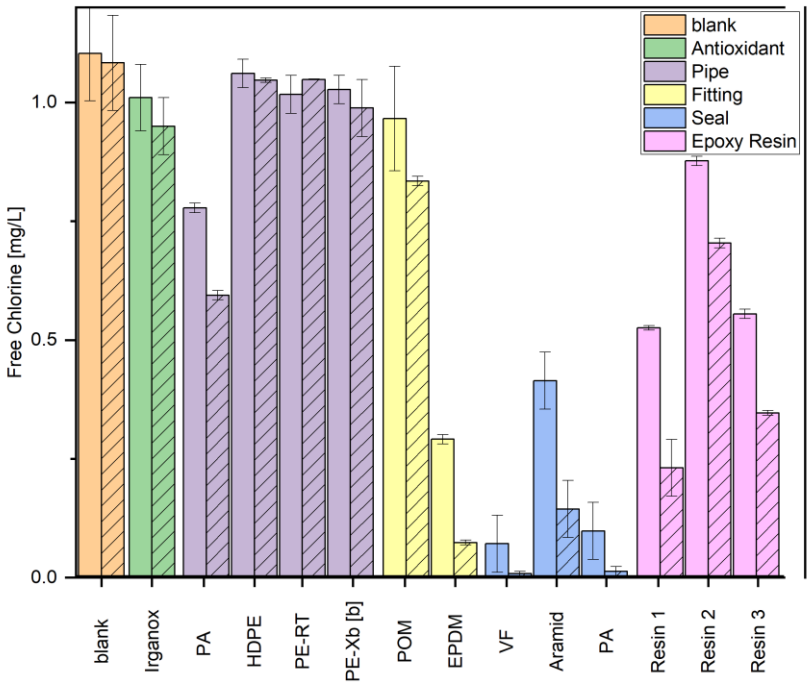

Figure 3 Free chlorine in migration water with selected powdered materials after 15 (non-shaded) and 60 min (shaded)

The results in Figure 4 indicate, that PE-Xc is more affected by ClO_2, PE-Xb by NaOCl, while PE-Xa is affected similarly by both disinfectants. An increase in ClO_2 concentration only leads to higher consumption in PE-Xa and PE-Xb.

The disinfectant consumption (ΔC_R) in migration tests was calculated as follows:

$\Delta C_R = C_{R,blank} - C_{R,exp}$

$C_{R,blank}$ is the residual disinfectant concentration measured in the blank samples and $C_{R,exp}$ is the residual disinfectant measured in the samples containing the test materials. All concentration values are expressed in $mgCl_{2\text{-}eq}/L$. ΔC_R is only caused by the presence of the tested materials and not to the degradation of the disinfectant. Tests were also carried out with PE-X pipes and higher disinfectant concentrations (NaOCl at 50 $mgCl_2/L$ and ClO_2 at 50 and 19 $mgCl_{2eq}/L$). The results are shown in Figure 4.

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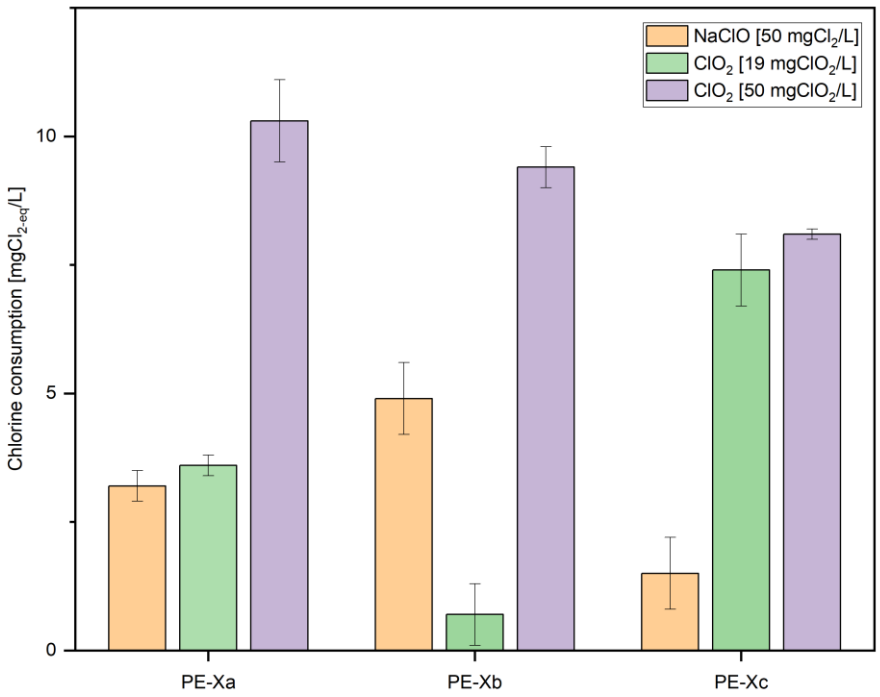

Figure 4 Consumption of chlorine (ΔC_R) expressed as Cl_2-eq, in the migration tests of PE-X Pipes in powdered form

Comparing the results of NaOCl at 50 mg/L Cl_2 and ClO_2 at 19 mg/L dosed to have the same oxidising power, PE-Xc is more affected by ClO_2 and PE-Xb by NaOCl, while PE-Xa is similarly affected by both disinfectants. An increase in the ClO_2 concentration only leads to higher disinfectant consumption for PE-Xa and PE-Xb.

The influence of the outside temperature on ΔC_R was also analysed. The DIN EN 12873-1 (2014) specifies a temperature range of 23°C ± 2°C. Therefore, migration water tests with selected pipe materials and an AO were carried out at 21°C and 25°C ± 0.5°C as end points of these temperature ranges. The contact time was 72 h. The influence of temperature on chlorine consumption has already been highlighted in a model by Ma et al. (Ma, 2021). A slightly increased chlorine consumption (22% on average) was determined for all samples analysed at elevated temperatures as shown in Figure 5. EPDM showed an almost complete chlorine consumption at both temperatures.

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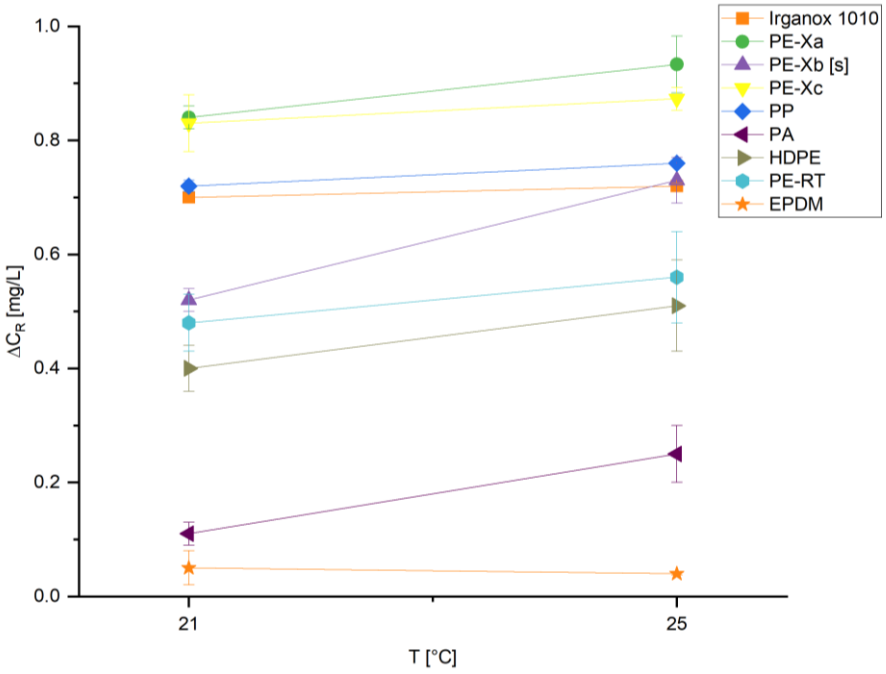

Figure 5 Consumption of chlorine (ΔC_R) expressed as Cl_2-eq in the migration tests with selected pipe materials (700 mg/L) and Irganox 1010 at different temperatures.

Furthermore, the disinfectant decay for cement mortar tubes was tested. For this the post-ageing migration stage was introduced to evaluate the effect of a prolonged contact of pipe materials with disinfectant during the ageing stage. As can be seen in Figure 6, this was done comparing the decay of disinfectant during migration and during post-ageing. Even if the duration of each step was 72h, re-chlorination was performed every 24h. Focusing on Figure 6a, the decay of NaOCl does not slow down within the migration or the post-ageing step. On the contrary, comparing the decay in the two stages, there is experimental evidence that in the post-ageing the decay is reduced. In Figure 6b, the same type of result was obtained, even if the difference between migration and post-ageing was less significant. The reason of the reduction of decay between migration and post-ageing could be that most of the TOC is released in the pre-conditioning and migration stages. Hence, if there is less TOC leached from the pipes, the disinfectant consumption is reduced.

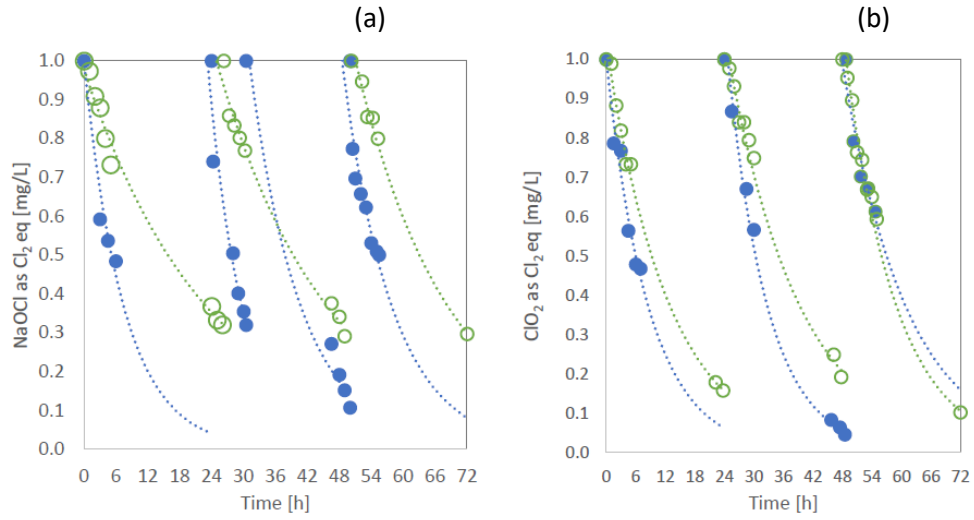

Figure 6 Disinfectant decay in cement mortar pipes: NaOCl (AI=11) (a) and ClO_2 (AI=11) (b) concentration in migration step 8 (blue dots) and post-ageing step 23 (green circles).

This project has received funding from the European Union's Horizon Europe research and innovation programme under grant agreement No 101081980.

Natural organic matter characterization

It was found that both chlorinated and non-chlorinated filtrated migration water samples showed an increase in DOC for all materials during the 72 h contact time compared to blank samples, which is illustrated in Figure 7.

The DOC concentration indicate that organic matter leached from the materials into the water. The samples of epoxy resins and PA caused the highest DOC concentrations. The chlorinated samples had a higher (20%) average DOC leaching than the non-chlorinated samples. However, the order of DOC concentrations for the materials was the same for both tests. Liu et al. (Liu, 2022) tested different microplastics (MP) where PE showed the highest resistance to fragmentation while the fragmentation during chlorination increased with the reaction time and could contribute to a higher DOC in chlorinated samples.

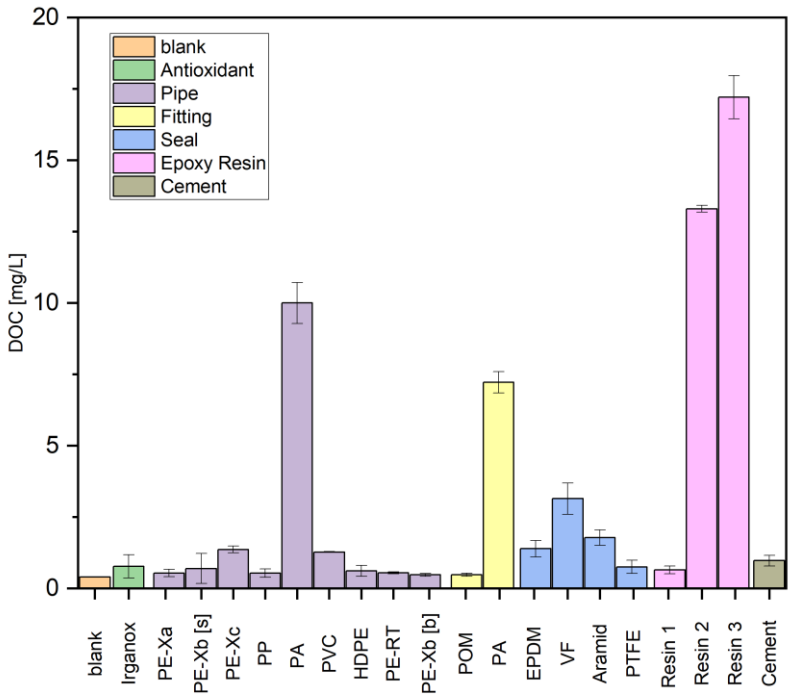

Figure 7 DOC concentration in the filtered migration water samples of the different materials categorised into groups in powered form (700 mg/L)

An increase in DOC was measured for higher concentrations of powder. Migration water samples with 50, 350 and 700 mg/L of powder were measured. In addition, the DOC increase in migration water samples was significantly correlated with the reaction time.

The leaching of DOC from different materials could be influenced by the composition, the polymer type and surface properties. In addition to the age of the plastic, it was found that old plastic leached more DOC than newer plastics under the same test conditions (Parveen et al., 2024). PET, for example, is susceptible to the hydrolytic cleavage of ester bonds, which can lead to the formation of carboxylic acids through the reaction in water (Hawkes et al., 2019).

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Figure 8 shows for the comparison between powder and tube tests that an average 32% increase in DOC was shown for migration water with powder.

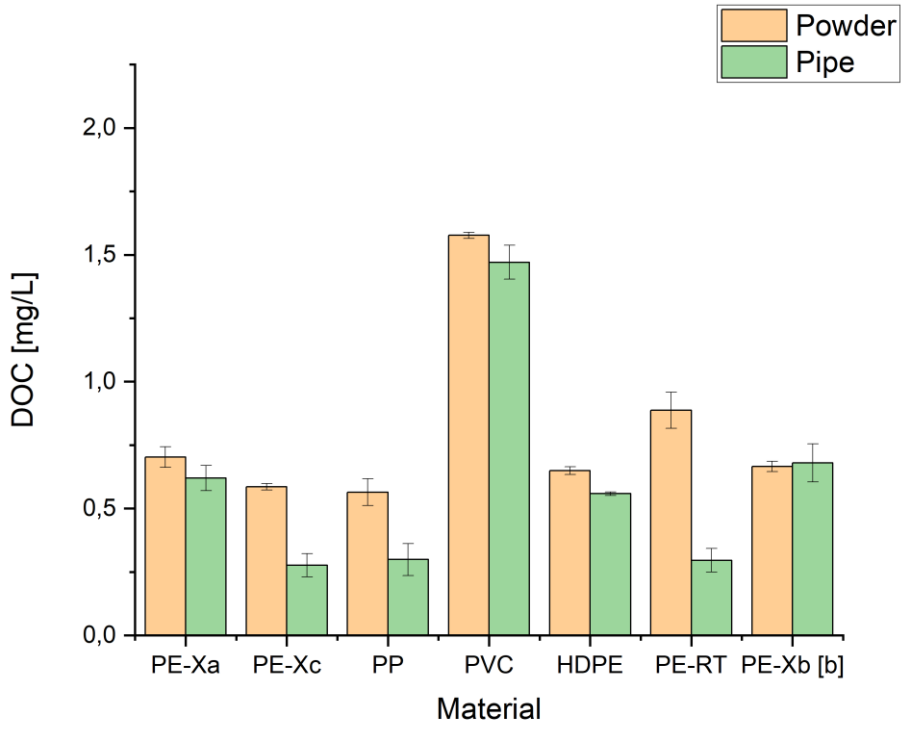

Figure 8 DOC concentration in the filtered samples of the different materials in powered form and in pipes in comparison

TOC release from cement mortar lined pipes is most likely due to the use of organic additives across the production chain. Figure 9Figure 9 Release of TOC along with experimental steps and in different stages: Pre-conditioning (steps 1-4; green), Washing (step 5; orange), Migration (steps 6-8; purple). shows the leaching of organic matter in pre-conditioning, washing and migration. After the first two steps of pre-conditioning with greater releases, no specific pattern can be seen in the other steps and stages.

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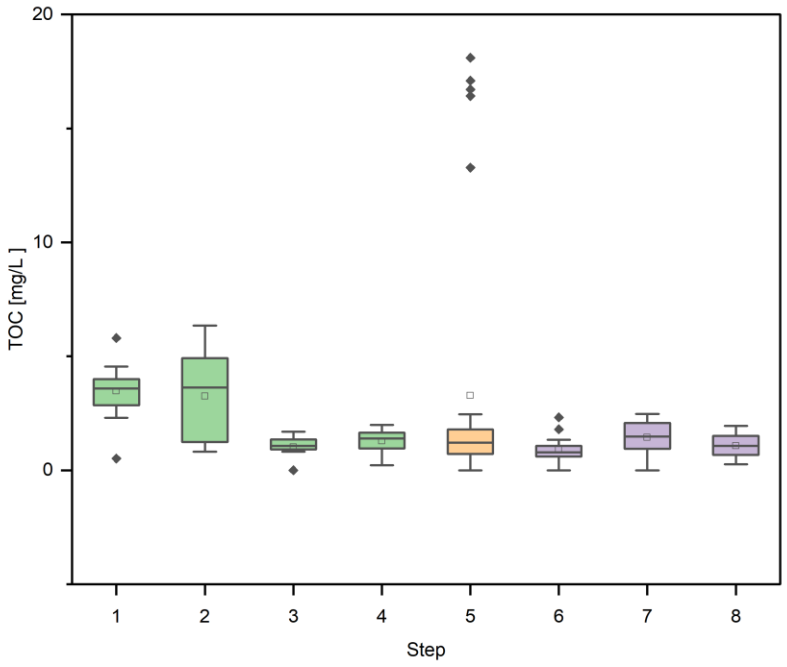

Figure 9 Release of TOC along with experimental steps and in different stages: Pre-conditioning (steps 1-4; green), Washing (step 5; orange), Migration (steps 6-8; purple).

Leaching in the first three steps seems to be promoted by the presence of the disinfectant (Figure 10a). The greatest release is found with ClO_2, followed by NaOCl and without the disinfectant as previously reported by Młyńska et al. (Młyńska, 2019). The effect of the water matrix on Figure 10b is also noteworthy. In steps 1 and 2, there is a notable reduction in leaching when the standard water is used, in comparison to type 2 and type 3, where the leaching is almost identical. Liu et al. (2022) reported that hardness and alkalinity can affect the leaching substances in drinking water. Alkalinity higher than 30 mg/L $CaCO_3$ may slow down the corrosion of cement mortar in addition to the pH due to the formation of a protective precipitation layer.

Three types of water were all characterized by an alkalinity higher than 30 mg/L $CaCO_3$, however, the type 3 had an alkalinity more than two times higher than the other two. Since type 3, together with type 2, showed the most release, it is not clear if the precipitation layer may reduce the leaching of only metals, such as aluminium, or if it can affect also the release of TOC.

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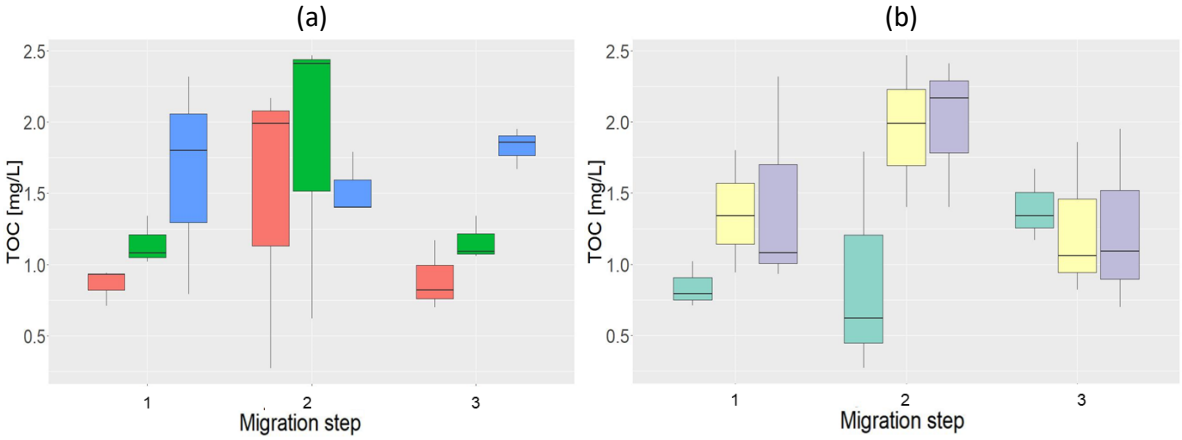

Figure 10 TOC release in migration steps. (a) Comparison of disinfectant conditions. From left to right: no disinfectant (red); NaOCl (green); ClO_2 (blue). (b) Comparison of type of water. From left to right: Standard (aquamarine); Type 2 (yellow); Type 3 (violet).

No significant differences were found in absorption spectra of water samples that contained powdered material and had not come into contact with disinfectants and those that had come into contact with NaOCl compared to blank samples. In water samples with powder that were in contact with ClO_2, an increase in absorption in the wavelength range from 240 to 270 nm was observed compared to the blank samples and is shown in Figure 11 and Figure 12 for PE-Xa. The increase was between 5-20%. The increase might be explained by the leaching of alkylphenols, ketones and aldehydes, which absorb in this wavelength range (Korshin et al., 1997; Papciak et al., 2020).

LC-OCD chromatograms provide more information about the composition of the organic matter, regarding molecular weight. It was observed that a significant increase in the organic carbon content of all tested materials was only observed when they came into contact with ClO_2-disinfected water. Figure 11 shows examples of the chromatograms for the three epoxy resins tested without disinfectants and a blank sample as a reference one. The increase in dissolved organic carbon content be associated mainly with low molecular weight acids and low molecular weight neutral compounds according to the classification by Huber et al. (2011). For the fluorescence data, no significant differences were found between the blanks and the migration water samples.

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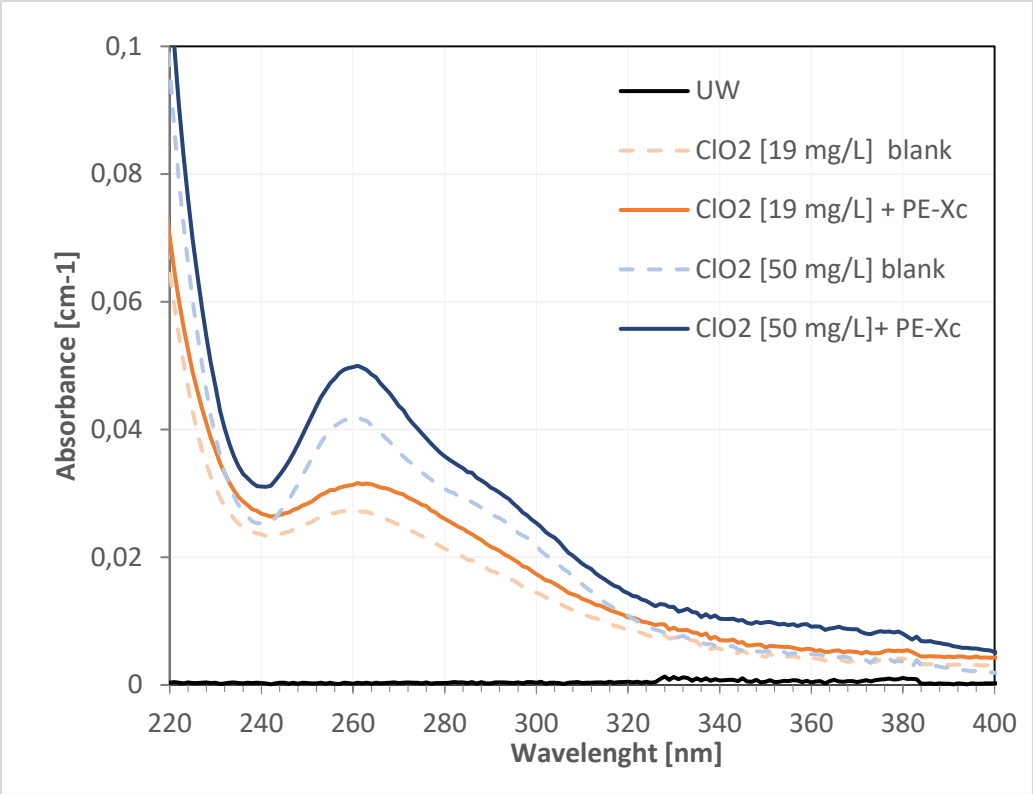

Figure 11 Absorbance spectra, evaluated between wavelength 220-400 nm for migration waters from PE-Xa spiked with ClO_2 [19, 50 mg/L] and ultrapure water (UW) as reference.

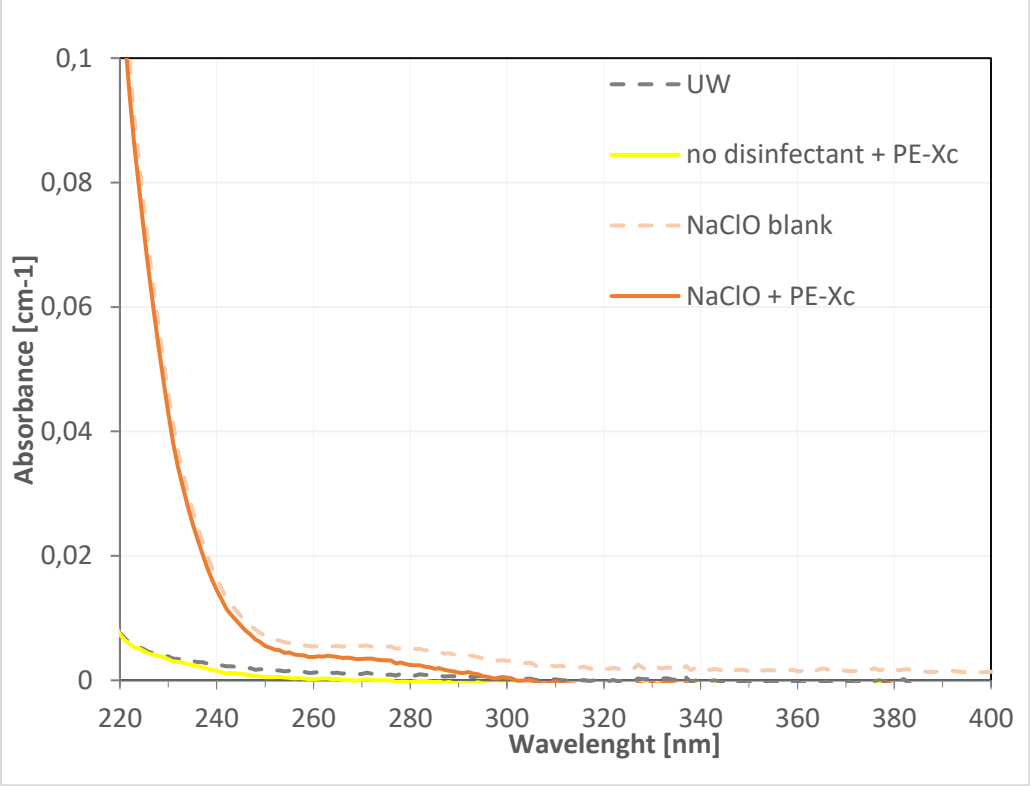

Figure 12 Absorbance spectra, evaluated between wavelength 220-400 nm for migration waters from PE-Xa spiked with NaOCl [50 mg/L] and ultrapure water (UW) as reference

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Disinfection by-product formation

In all samples, trichloromethane (TCM) and dichloroacetic acid DCAA were detected as conventional regulated DBPs formed by NaOCl and ClO_2. The amounts of TCM in migration waters from tests with powdered materials are shown in Figure 13. As expected from literature, TCM originated mainly from samples spiked with NaOCl (Padhi et al., 2019; Wang et al., 2021). The presence of TCM in NaOCl blank samples is likely due to the storage of the disinfectant in a PVC bottle.

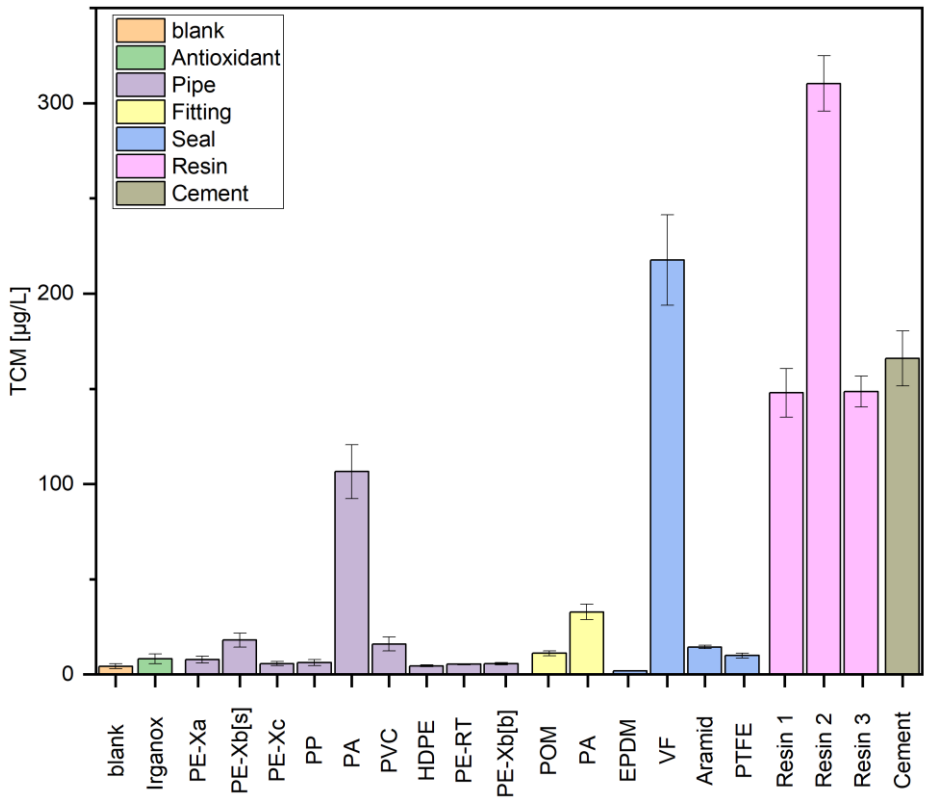

Figure 13 The amount of trichloromethane (TCM) in migration water samples after contact times of 72 h, 700 mg/L powdered materials and disinfectant doses of 1 mg/L Cl_2 (NaOCl)

With concentration of above 100 µg/L, the TCM formations were highest in the migration waters of the epoxy resins and cement mortar. The TCM concentration for VF of 217 µg/L was more than fifteen times higher than for other sealing materials. PA with an average TCM concentration of 106 µg/L caused by far the highest value of TCM among the pipes. The other pipes caused TCM concentrations in the range of 5 to 18 µg/L. Small amounts of TCM (3.2-5.5 µg/L) were also detected in ClO_2-disinfected samples. In some measurements, samples spiked with NaOCl and with powder were found to have a lower TCM concentration. This occurrence is likely due to the potential absorption on the surface of pipes described by (Cao et al., 2020) and further investigation is required to understand the potential desorption of volatile DBPs.

In addition, bromodichloromethane (BDCM) was found in the migration water sample of VF and dichloroacetonitrile (DCAN) in migration water sample of VF and aramid. Chlorobenzene was found in migration water samples of PP and PA pipes as well as from VF and resin 2.

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The THM contents in migration test samples using the new method with powder and in the standardised form are summarised in Figure 14. The two tests were run simultaneously in the same fume cupboard at the same room temperature (23°C). On average, the value for powder tests was 22% higher than the values from tests in tube form. Only with PE-Xc was the TCM value in the pipe increased compared to the powder.

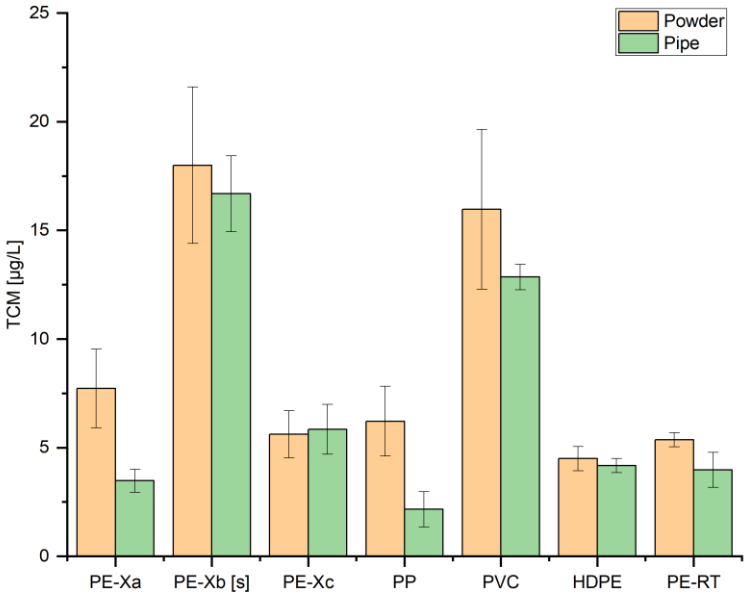

Figure 14 Amount of TCM in migration water samples by material in comparison of the two different test methods.

Analogous to the THM measurements, only one species, dichloroacetic acid (DCAA), was detected in almost all samples when analysing the migration water samples for the eight HAA. The range was from 0.25 to 6.2 µg/L. When comparing the THM and HAA measurements, it is noticeable that the highest values were found for VF and PA. Overall, however, the values for TCM were significantly higher than those for DCAA, averaging over 20 times higher values, similar findings have also been documented in other studies (Golea et al., 2017; Parveen et al., 2024). The comparison can be seen in the Figure 15.

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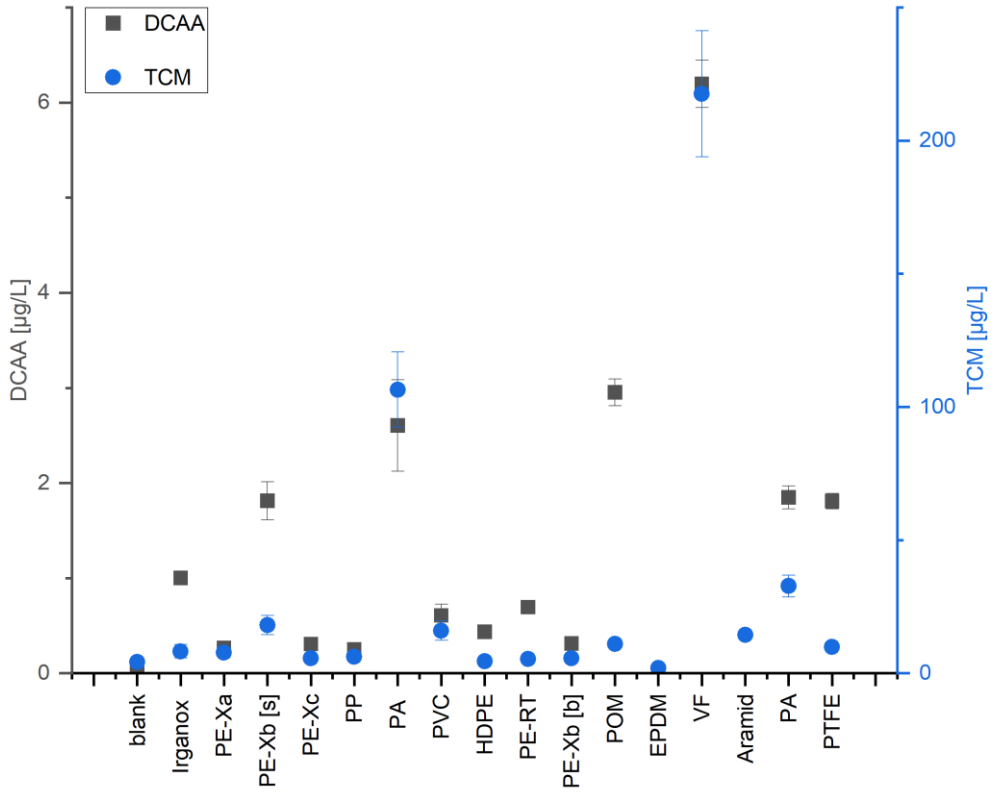

Figure 15 Concentrations in µg/L of DCAA (left y-axis) and TCM (right y-axis) in migration waters of different materials

A comparison of the two methods in Figure 16 shows, that the formation of HAAs (DCAA) is increased in the tests where the pipe material was present as a powder compared to the conventional method. On average, the values were 39% higher.

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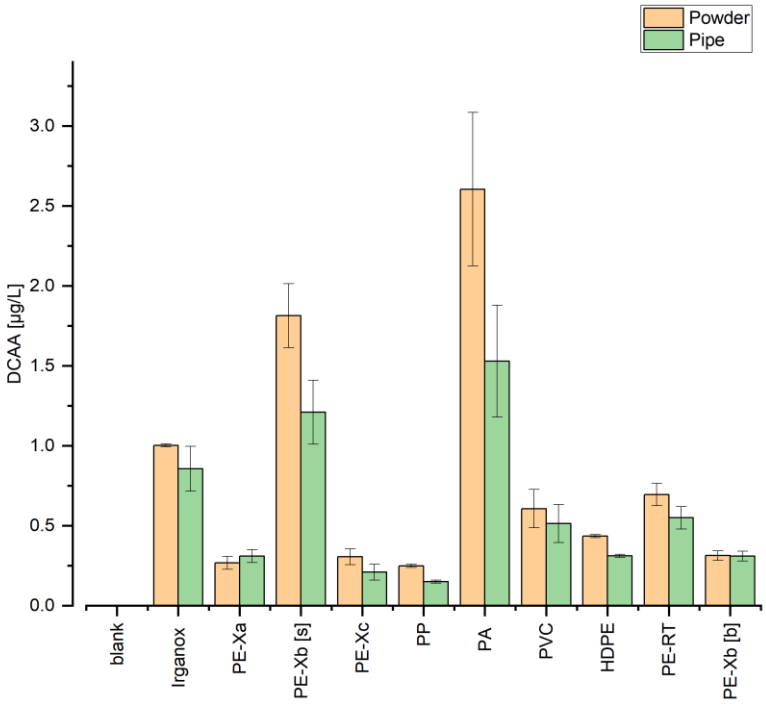

Figure 16 DCAA concentrations in the migration waters where pipe materials were present in powder form compared to conventional material tests.

The results for the sulfonic DBPs from chlorinated migration water of pipe material in powder form are demonstrated in Figure 17 and show that dichloromethanesulfonic acid (Cl_2MSA) was more detected in PVC and PE-Xb materials.

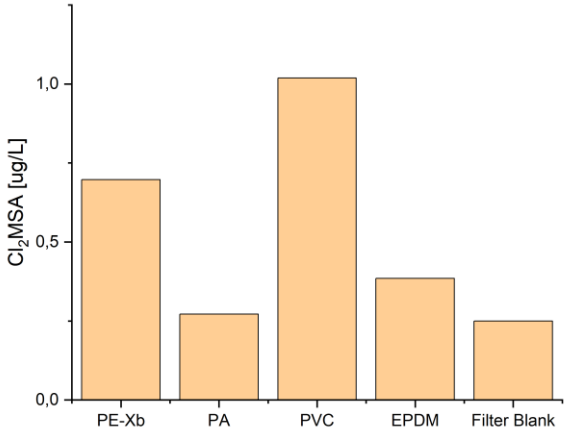

Figure 17 Dichloromethanesulfonic acid (Cl_2MSA) concentration in migration water samples from different powdered pipe materials.

The measured sulfonic DBPs differ from the results for THMs and HAAs, where more were measured in PE-Xb and PA, and fewer in PVC. However, Cl_2MSA was also found in the filter blanks samples. From this, it can be concluded that there might be the precursors of these DBPs in the PTFE filter used, which must be considered in future experiments.

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Bromochloromethanesulfonic acid, chloroacetonitrilesulfonic acid, dichloroacetonitrilesulfonic acid, chloroacetamidesulfonic acid, dichloroacetamidesulfonic acid, and dichloroacetaldehydesulfonic acid could be detected by SFC-QTOF analysis after freeze-drying enrichment, in all migration water samples, but not quantifiable. Chloroacetamidesulfonic acid was only found in PVC migration water samples.

Conclusion

The newly applied method of cryogenic milling of materials approved for drinking water into powder enables a standardised procedure for the analysis of migration waters from different product types. By flexible dosing of the powder, different S/V ratios can be adjusted in the migration tests, whereby higher concentrations of powder have shown higher organic matter and DBP contents in the analysed samples.

Furthermore, the tests with powdered material proved to be more sensitive for detecting DBP formation compared to conventional methods. The study suggests that additional research can optimize testing protocols and ensure safer material choices for drinking water systems.

In the organic matter characterization, DOC appeared to be the clearest indicator as a sum parameter for leached substances. In addition, higher DOC releases from the materials led to both higher chlorine consumptions and higher DBP formations and a correlation between these factors can be observed.

The results indicate that plastic pipes have a limited impact on DBP formation compared to other materials in contact with drinking water. Rubber seals and cement mortars, however, exhibited significant contributions to chlorine consumption and organic matter leaching. Epoxy resins were found to have the most substantial effect on water quality, particularly in the formation of trichloromethane (TCM) and other DBPs.

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