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Investigation of organic compounds migration from polymeric pipes into drinking water under long retention times

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Abstract

Unplasticized polyvinyl chloride pipes (uPVC), Polypropylene random pipes (PPR) and Polyethylene pipes (PE) for drinking water were tested with respect to migration of total organic carbon (TOC) into water. The investigation was carried out under different long retention times aiming to investigate TOC migration trends of different polymeric pipes over time and the corresponding water quality changes like chlorine decay. Experiment results showed TOC release from all pipes increased significantly over time. PE pipe showed the highest TOC concentrations among all tested pipe materials in this study. Chlorine consumption data observed in this research followed the shape of TOC values.

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Keywords: Polymeric pipes; drinking water; migration; TOC; retention time; chlorine

1. Introduction

Polymeric pipes with many advantageous attributes, such as long durability, corrosion resistance, low cost, easy installation, etc., have been increasingly used as substitutes for old metallic pipes in drinking water systems. During this development, various types of plastic pipes made of polyethylene (PE), polyvinylchloride (PVC) and polypropylene (PP) are predominantly presented in the potable water distribution networks all over the world (Bucheli-Witschel et al., 2012, Löschner et al., 2011, Trew et al., 1995, Zhang et al., 2011). According to an

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estimation from U.S Environmental Protect Agency, the United States will spend between \$20 and \$22 billion over the next 20 years to repair and replace its buried infrastructure in drinking water systems, while in north America the two most popular polymeric materials are PVC and PE (Whelton and Nguyen, 2013). In China, in the light of the 10th five-year national plan, plastic pipes with a nominal diameter (DN) below 400mm were proposed to be employed in urban drinking water networks and accounted for about 80% of all the pipe materials used in water distribution systems by 2015 (National Chemical Building Materials Committee, 2000). Thus, polymeric materials are increasingly becoming the important link between the water plant and the customer's tap.

However, one critical concern facing polymer materials should be very well aware that unexpected migration compounds might present in the water corresponding with plastic piping. This problem is likely attributed to the vast introduction of various monomers, antioxidants, catalysts, colorants, solvents, fillers, lubricants, etc. with in the pipe production processes. Although mechanical and physicochemical properties of the pipes are admirable, the high occurrence that unwanted organic and inorganic compounds leaching from polymeric pipes into the drinking water resulting in contamination would be observed. Migration of contaminants from polymeric pipes could occur directly by diffusion of chemical components within pipe materials into the water, or indirectly by, for example, decomposition products of additives used in pipe manufacturing and by-products formed by reaction among compounds in water (Adams et al., 2011, Brocca et al., 2002, Skjevraak et al., 2003, Walter et al., 2011). At present, a number of papers have been published focusing on the contaminant migration from PVC and PE pipes, with some effort toward PP materials. Extensive researches have been carried out to show that the main leachates from PVC pipe are metal stabilizers like lead, tin, barium, calcium and etc. (Adams et al., 2011, Al-Malack, 2001, Lasheen et al., 2008), vinyl chloride monomers (Al-Malack, 2004, Al-Malack and Sheikheldin, 2001, Al-Malack et al., 2000, Walter et al., 2011) and other contaminants related with plasticizers, antioxidants and lubricants commonly used in pipe manufacturing processes (Heim and Dietrich, 2007, Löschner et al., 2011, Skjevraak et al., 2003). As less metallic stabilizers are used in PE pipe manufacturing (Zhang et al., 2011), contaminants detected in waters in contact with PE pipe are almost organic compounds, especially volatile compounds (VOCs) mainly related with antioxidants, their degradation products and solvents (Brocca et al., 2002, Durand and Dietrich, 2007, Löschner et al., 2011, Lund et al., 2011, Skjevraak et al., 2003). In contrast to PVC pipe, there's no consensus as to which contaminants are most common in PE pipe due to the limited data available and experimental design deficiencies in the laboratory testing section (Whelton and Nguyen, 2013). However, few studies were found that characterized migration from PP pipe. Löschner et al. (2011) discovered that the significant substances leaching from PP were 2, 4-di-tert-butylphenol and 3, 5-di-tert-butyl-4-hydroxybenzaldehyde, which were degradation products of antioxidants used as additives for the plastic materials. Additionally, Hametner (1999) studied the influence of the migration of phenolic antioxidants, which are typically used for the stabilization of PP pipes, on the quality of drinking water.

The contaminant migrations from polymers have been usually investigated by applying indirect and direct methods (Whelton and Nguyen, 2013). The indirect methods involve monitoring TOC concentration, pH, disinfectant level, and taste and odour attributes, while the direct methods involve chromatographic and spectroscopic separation of organic and inorganic compounds in the water. Thus, various indicators under direct and indirect methods were called direct indicators (e.g., organotins, phthalates, phenolic antioxidant, monomers) and indirect indicators (e.g., TOC, pH, total chlorine, odour intensity, turbidity), respectively. Notably, indirect and direct indicators are related with each other and both indicate the leaching behaviour of polymer pipes (Bucheli-Witschel et al., 2012, Löschner et al., 2011, Skjevraak et al., 2003).

Organic compound release from polymers reported as TOC concentrations has been discussed by many investigators. Qu et al. (2012) carried out tests of TOC migration to distilled water, dispensed water and nutrient solution for three brands of high-density polyethylene (HDPE) irrigation pipes. Results showed that TOC migrating from different brands of pipes tended to improve with increased temperature during all of test periods. Löschner et al. (2011) found that the direct method by gas chromatography-mass spectrometry (GC-MS) cannot detected all leachable organics and TOC as an indirect indicator can somehow make up the defect and give an overall explanation of the organic compounds occurring in the migration waters. Heim and Dietrich (2007) examined leaching of organic compounds, measured as TOC, from HDPE and chlorinated polyvinyl chloride (cPVC) pipes. Results showed that HDPE and cPVC pipes both showed increases in TOC and decreases in

disinfectant residual. Water samples contacted with HDPE produced 0.14 ug TOC/cm^2 pipe surface, which was significantly greater than the TOC increase from cPVC ($0.08 \text{ }\mu\text{gTOC/cm}^2$), whilst HDPE showed more disinfectant demand than cPVC and was found to contribute substantial odor. The disinfectant consumption can likely be attributed to the interaction of oxidizing disinfectant species with organics from polymers. Research from Skjevraak et al. (2003) also measured TOC but the amount leached was below their instrumental detection limit of 0.4 mg/L . Bucheli-Witschel et al. (2012) investigated TOC values of different polymers in contact with drinking water and gave an evaluation for those commercial polymers according to the requirements for TOC from migration tests in German. As a summary, TOC can represent the overall information of the total organic contaminants migrating from polymers and is easily affected by environmental conditions and significantly correlated with water quality problems such as odour and disinfectant consumption and used as controlled parameter in water regulations as well.

Within a water distribution network, the flow status differs in pipe sections. The flow velocity is normally greater in the trunk pipes than the one in service lines. Thus, different retention times occur in different pipe sections and result in different flow status. Water in some pipes sections with a high flow velocity may renew fast. In contrast, long retention time might appear at the dead end of the system or on service line where there has low residence rate or household leaves for a long vacation. It has been commonly noticed that long retention time could result in water quality problem in the water distribution system, especially low concentration of chlorine residual. Past literatures have reported the investigation on impact of stagnation time on migration of some specific substances from water pipes (Lasheen et al., 2008, Lund et al., 2011, Lytle and Schock, 2000, Walter et al., 2011). However, few studies researched the influence of various long retention times related to the stagnant conditions in real distribution system on water quality from the perspective of contaminant migration of different plastic pipes.

The objectives of this study were to (i) determine the characteristics of TOC migration from polymeric pipes under the condition of long retention times, and (ii) investigate the relationship between TOC migration and chlorine consumption.

2. Materials and methods

2.1. Polymer pipes used in this research

Three types of commonly used polymeric materials, i.e. uPVC, PE and PPR, were selected in this research and they all have drinking water contact approval in China. The dimension of uPVC pipes was $63 \times 3.00 \text{ mm}^2$ (external diameter \times wall thickness) with an inner diameter corresponding to 57mm. The ratio of the surface area (SA) of the tested pipe segments intended to come into contact with test water to the volume (V) of the test water, referred to as SA/V in the following text, were expressed per centimeter, i.e. cm^{-1} (which is cm^2/cm^3). The surface area to water volume ratio (SA/V) of tested uPVC pipes was 0.7017 cm^{-1} . Tested polyethylene (PE) pipes has a dimension of $63 \times 4.7 \text{ mm}^2$ with inner diameter of 53.6mm and $\text{SA/V} = 0.7462 \text{ cm}^{-1}$. Two brands of polypropylene random (PPR) pipes were tested with inner diameters of 51.4mm and 45.8mm. Thus, the calculated SA/V was 0.7782 cm^{-1} and 0.8733 cm^{-1} , respectively. All the tested pipes were unused prior to the migration tests.

2.2. Migration tests for polymer pipes

Methodologies used worldwide to evaluate leaching behaviour of drinking water pipes mainly focus on directly and indirectly determining the effect of a polymer on water quality such as taste and odour, TOC concentration, and microbiological growth potential (Brocca et al., 2002, Bucheli-Witschel et al., 2012, Heim and Dietrich, 2007, Lund et al., 2011, Skjevraak et al., 2003). The most common test methods include organoleptic testing, TOC concentration, and specific contaminant detection (Rogers et al., 2004). In China, migration procedures for sanitary evaluation of pipes are described in the national standard GB/T 17219-1998 : standard for safety evaluation of equipment and protective materials in drinking water system. According to different methods, the most used test conditions are (i) static immersion testing (Bucheli-Witschel et al., 2012), (ii) static pipe segments (Brocca et al., 2002, Durand and Dietrich, 2007, Löschner et al., 2011, Skjevraak et al., 2003), and (iii) water flowing

intermittently or continuously through a pipe (Adams et al., 2011). Moreover, test conditions may also differ in water exposure duration, preconditioning and initial water quality (e.g., pH, disinfectant level and organic level). Not all studies applied the same migration procedures or test conditions, thus making the comparison among data difficult. Notably, most of the published papers reported migration data for new polymer pipes under static conditions. However, the research of migration under dynamic flow status was rare and complicated. Consequently, in the present research, the static pipe segments method (ii) was adopted and the influence of fluid dynamic state was not investigated.

According to the migration tests described in the European standard EN 12873-1(2003), the material sample is brought into contact with water during three (or more) subsequent migration periods under stagnant conditions at a specific temperature and for a specific time span. The obtained migration water was then analysed for released TOC or taste/odour. However, in the Chinese national standard GB/T 17219-1998, migration test was carried out once for 24h contact time at 25 ± 5 °C and the multiple analysis of migration water include metals, taste and odour, some specific compounds and selective indexes. The former standard used several short duration periods to indirectly investigate the extent of migration, while the latter one were able to directly and indirectly study the influence of contaminant migration of pipes on water quality.

Based on the two regulatory standards, a continuous migration procedure with various retention times was developed in this research to determine the accumulation trend of TOC migrating from different polymeric pipes over time and investigate the influence of contaminant migration on the corresponding water quality.

Within this research, three types of plastic pipe were tested under the developed continuous migration procedure as follows. The test pipes were first cut into three pipe segments of the same length (30cm) as a set of pipe samples for each type of polymers. Prior to the migration test, pipe samples were pre-treated according to Chinese national standard GB/T 17219-1998: initially cleaned the pipes samples with tap water, filled the pipe segments with tap water and flushed constantly for 30 min, then rinsed with deionised water for 3 times. The test water used in this study was prepared using deionised water and pure chemical reagents (i.e., sodium hydrogen carbonate, Calcium chloride anhydrous and sodium hypochlorite) to get a simulated drinking water with 2mg/L total chlorine, 100mg/L calcium hardness, pH range 7.8-8.0 and TOC concentrations lower than 0.2mg/L. No additional TOC was added. These water quality values of test water were determined based on the mentioned values in GB/T 17219-1998. Immediately after the preconditioning, pipe segments were completely filled with freshly prepared test water and sealed using Teflon-lined stoppers at both ends and left under static conditions for continuous migration.

Unlike the migration time procedures described in EN 12873-1(2003) or GB/T 17219-1998, the continuous migration procedure in this study lengthened the stagnation time and extended migration tests under five separate long retention conditions. For each type of pipe material, five separate continuous migration tests with retention times of 24h, 48h, 72h, 144h and 240h, respectively were performed under stagnant conditions at 25 ± 5 °C. After each time of exposure under specific test conditions, the water containing the leachates, referred to as the migration water in the following test, was collected for further analysis with respect to TOC, total residual chlorine, pH etc. Moreover, negative control tests were created by placing the test water in 1000ml amber glass bottles containing Teflon-lined stoppers and left under the same test conditions (contact time, temperature and static status) as pipe segments. In addition, continuous migration tests for each of polymers were all carried out in triplicate.

2.3. TOC tests

TOC was measured as non-purgeable organic carbon (NPOC) using a total carbon analyser TOC-Vwp (Shimadzu, Shanghai). Migration waters were transferred into TOC-free vials (thread screw neck EPA vial, amber glass). Using phosphoric acid acidification, inorganic carbon in migration waters become carbon dioxide and was stripped by CO₂-free air. For each sample measurement in TOC-Vwp, three times of about 10ml were injected into the TOC analyser. Then, the TOC concentration in unit of mg/L was determined in the TOC analyser based on the chemical oxidation of organic substances using sodium persulfate solution. Finally, TOC concentration value of each sample was then the average of the three measurements. To prepare a calibration curve, potassium phthalate

(Guaranteed reagent, from Tianjin, China) was diluted with Milli-Q ultrapure water to obtain solutions with carbon concentrations ranged from 0.1 to 10 mg/L. The detection limit of the method was about 0.005mg/L.

In this experiment, all TOC concentrations of different polymers described in latter results and discussion section were obtained by subtracting the negative control TOC values from those measured in the migration waters from different polymer pipes.

Based on the TOC concentrations obtained from all continuous migration tests and the SA/V of tested pipe segments, TOC migration rates were calculated according to the following equation based on EN 12873-1 (2003):

$$m = ct^{-1} (SA/V)^{-1} \quad (1)$$

Where m is the corresponding calculated TOC migration rates, expressed in unit of $\mu\text{gTOC}/(\text{d}\cdot\text{cm}^2)$, c is the concentration of TOC in mg/L obtained in the migration water, SA/V is surface area-to-water volume ratio in cm^{-1} and t is the duration of the migration time in days.

2.4. Chlorine tests

For the chlorine tests, the total chlorine was measured by HACH® pocket colorimeter TM II analysis systems. Using appropriate powder pillows based on the DPD method ((N,N-diethyl-p-phenyldiamine), concentrations of total chlorine were determined either within low range (0.02-2.00 mg/L Cl_2) or within high range (0.1-0.8 mg/L Cl_2). Notably, total chlorine tests were conducted before and after the test water were stored in pipe segments and negative controls. Chlorine consumption in mg/L was calculated by subtracting negative control values from those obtained in the corresponding migration waters. Moreover, chlorine consumption rate in mg/ (L·d) were obtained by dividing chlorine consumption values by the duration of migration time in days (d).

3. Results and discussion

3.1. Determination of TOC migrating from uPVC, PE and PPR

The characteristics of TOC migration obtained from different types of pipe materials were presented in Fig.1.

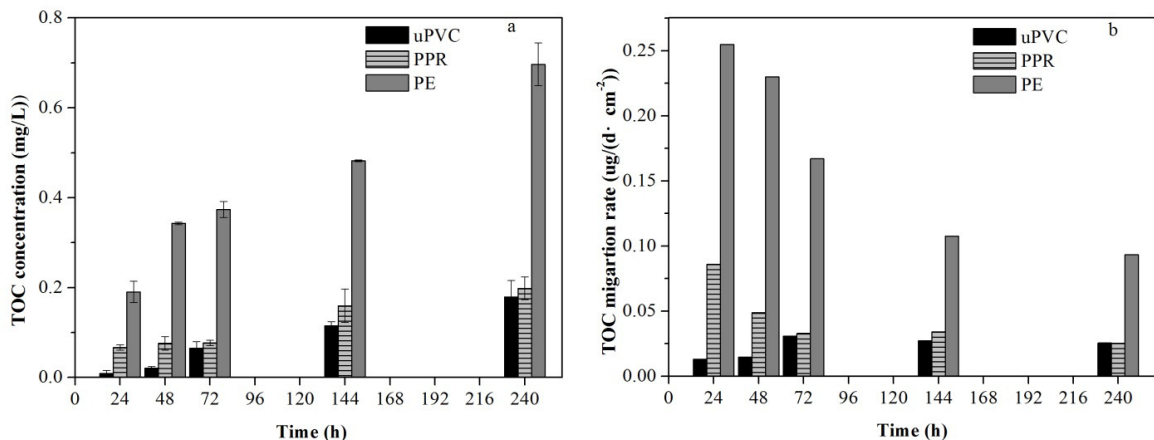


Fig. 1. (a) TOC concentrations (mg/L) and (b) TOC migration rates ($\mu\text{gTOC}/(\text{d}\cdot\text{cm}^2)$) determined in migration waters of uPVC (black columns), PE (grey columns) and PPR (light grey columns filled with horizontal lines) pipes under continuous migration method. Error bars in Fig.1 (a) show standard deviation across triplicate samples.

As shown in Fig.1 (a), the concentrations of TOC migrating from all plastic materials had a significant increasing trend with the increment of retention time, suggesting that under long stagnant conditions organics migration from plastic pipes would accumulate fast and likely deteriorate the water quality. For PE, TOC concentrations showed a rapid increase from 0.19 mg/L to 0.34 mg/L by 48 h of exposure. This situation may be attributed to the presence of some residual organic compounds in the surface of pipes. There was a slight increase of TOC concentrations in the waters from 48 h and 72 h of exposure. The second significant increase stage of TOC was seen to take place after 72 h of exposure. By 10 days of stagnation, TOC concentration in migration water of PE was about 0.7mg/L. Unlike PE, water in contact with PPR presented a gradual increase of released TOC. After 240 h of exposure, TOC concentrations in migration waters from PPR were about 0.2 mg/L. For uPVC pipe, the amount of TOC in migration waters showed a slow but steady increase over stagnation time. After 48 h of stagnation, TOC concentration from uPVC was at a small level of about 0.01 mg/L, and by the end of the experiment (240h), it increased to 0.18mg/l. Overall, PE showed the highest amount of TOC release during all migration tests. The corresponding values were about 2-21 times higher than for other materials. Additionally, TOC migrating from PE increased faster over stagnation time than the other pipe materials, which would be further explained by the corresponding TOC migration rates.

Clearly in Fig.1 (b), TOC migration rates from different polymers differed with respect to the magnitude and the trends over stagnant time. TOC migration rates of PE pipes presented a drastic decline by 72 h of stagnation and then stabilized at a level of about $0.1 \mu\text{gTOC}/(\text{d}\cdot\text{cm}^2)$. For PPR pipes, TOC migration rates went through an initial high-level of $0.09 \mu\text{gTOC}/(\text{d}\cdot\text{cm}^2)$ down to a plateau of about $0.03 \mu\text{gTOC}/(\text{d}\cdot\text{cm}^2)$ with the increasing retention times. However, TOC migration rates of uPVC were observed with a small range of fluctuation over retention time, which remained at a small range of $0.01\text{-}0.03 \mu\text{gTOC}/(\text{d}\cdot\text{cm}^2)$. Generally, the significant decrease of TOC migration rates over stagnant time obtained from PE and PPR pipes implied that intensity of organics migration from polymers would decay over time till an equilibrium between polymer and water was established.

In summary, increasing retention times of water in contact with pipes would promote the migration of organic compounds from polymers. As will be shown later, more organics migration from different polymers corresponded to more chlorine demand in migration waters. Moreover, taking into account the TOC concentrations and TOC migration rates observed from different retention times in this research, the following order of materials was obtained: PE>PPR>uPVC. This situation could be attributed to the chemical compounds used in pipe manufacturing differed a lot among different pipe materials. Generally, there are more organic additives related with antioxidants used in the manufacturing of PE and PPR pipes compared to uPVC pipe (Zhang et al., 2011). Leaching of organic compounds as TOC from pipe materials to water has been demonstrated in previous studies. TOC concentrations of PEX pipe, PB and plasticized PVC pipes were shown to decreased over migration periods (Bucheli-Witschel et al., 2012). The amount of TOC leached from either HDPE or cPVC pipes did not change with subsequent flushes and was not affected by the presence of a disinfectant (Heim and Dietrich, 2007).

3.2. Residual chlorine over time

Total chlorine was measured before and after the migration tests both in migration waters and negative controls. Based on those measured total chlorine concentrations, the amounts of chlorine consumption and the chlorine consumption rates were calculated. During the time frame in this research, total chlorine concentrations of all negative controls remained at the initial designed value of 2mg/L in fresh test water, which may indicate that within at least 10 days of stagnation, chlorine was not consumed by the test water with a very low TOC value and low ion concentrations. Thus, the observed variability of total chlorine concentrations before and after migration tests for each pipe material may be attributed to the chemical reaction between chlorine and contaminants leached from polymer pipes into drinking water. The chlorine consumption data collected during continuous migration tests were shown in Fig.2.

As seen clearly in Fig.2 (a), the amount of chlorine consumption of all polymers presented a significant increase over stagnant time. Notably, the highest amount of chlorine consumption were consumed in the migration waters from PE pipes. By 10 days of stagnation, total chlorine consumed in migration water from PE was about 1.9 mg/L, which was very close to the initial value (2 mg/L) of fresh test water. This implicated that under long stagnation

conditions chlorine consumption would be enhanced resulting in insufficient residual chlorine in the piping water, finally causing a microbial problem in drinking water. Overall, the amount of chlorine consumption observed from uPVC and PPR were approximately 1-3 times lower than from PE. Meanwhile, water in contact with PPR pipe exerted more chlorine consumption than with uPVC pipe. After 10 days of exposure, the concentrations of total residual chlorine in migration waters from uPVC and PPR were 0.36 mg/L and 0.55 mg/L, respectively. These indicated that under such long retention time, compared to PE pipes, the risk of water quality problems caused by uPVC and PPR pipes appeared to be much lower. In contrary, for chlorine consumption rates of different pipe materials, a decreased trend was clearly shown in Fig.2 (b). This might be attributed to two possible reasons. One was that as the amount of chlorine consumption increased over time, residual chlorine in migration waters decreased with time and thus lowered the reaction rates between chlorine and contaminants in water. The other reason was ascribed to the influence of TOC migration from polymers. Despite the increasing trend of TOC concentrations over time, the corresponding TOC migration rates declined, suggesting that less amount of chlorine would be consumed by organics per unit of time.

In general, considering the amount of chlorine consumption and chlorine consumption rates observed during each time of exposure, an order of materials tested in this research was obtained: PE>PPR>uPVC, corresponding to that obtained for TOC values (concentrations and migration rates) in Fig.1. This suggested that the disinfectant consumption can likely be attributed to the interaction with organics migrating from polymers. This chemical relationship might be linked with the production of disinfection by-products (DBPs), but they were not investigated in this research.

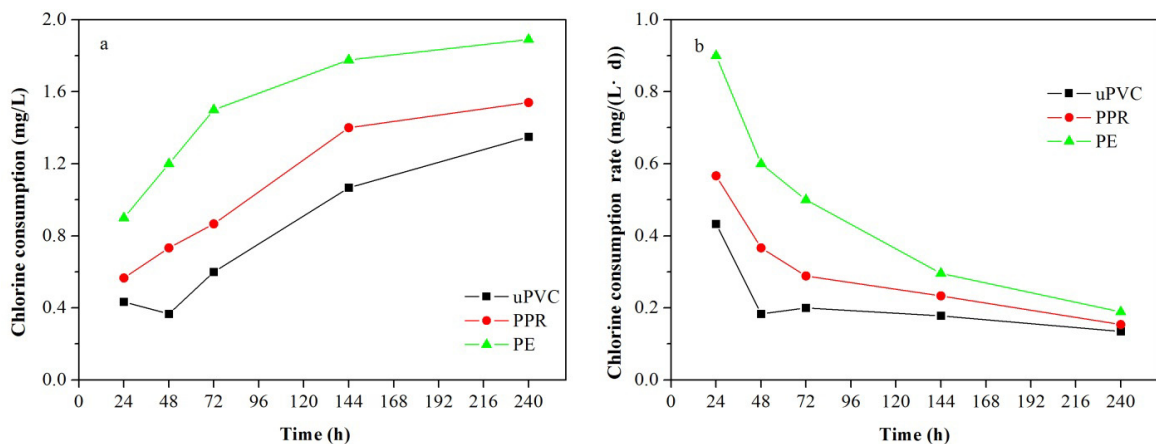


Fig. 2. (a) chlorine consumption (mg/L) and (b) chlorine consumption rates (mg/ (L·d)) obtained during continuous migration tests. Chlorine consumption rates mean the amount of total chlorine consumed in the migration waters per unit of time in days.

4. Conclusions

The continuous migration tests of this study indirectly investigated the characteristics of TOC migrating from three types of commonly used polymers under the conditions of long retention times and also showed the potential relationship between organics migration and water quality change like chlorine consumption.

Results showed that increasing retention times of water in contact with pipes would promote the migration of organic compounds from polymers and the levels of TOC release differed with different plastic materials. In this research, the order of materials with respect to the total amount of TOC migrating into water was observed: PE> PPR > uPVC. Chlorine consumption data observed in this research followed the shape of TOC migration values,

which allows to get a deeper insight into the migration process. The disinfectant consumption can likely be attributed to the chemical interaction with organics migrating from polymers through production of disinfection by-products, but they were not investigated in this research. Generally, under long stagnation conditions the drinking water quality could deteriorate fast with the growing migration of organic compounds and the dramatic consumption of chlorine to a very low level.

Overall, this study presented a valuable investigation of the organics migration as TOC from different polymers, which might alert industry to change the manufacturing process with less use of organic additives and finally to improve the quality of polymeric pipes in contact with drinking water. Further research should be carried out to investigate the DBPs formation related with organics migration, to study other potential factors like water quality, dynamic flow state and environmental conditions affecting the migration process and the possible migration mechanisms of organics leachates.

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