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# Surface analysis of pilot distribution system pipe autopsies: The relationship of organic and inorganic deposits to input water quality

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#### ABSTRACT

Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) surface analysis was conducted to characterise deposits in polyethylene pipes used in a novel pilot water distribution system (PDS). The system consisted of four (4) parallel distribution systems receiving water from different treatment processes, ranging from conventional coagulation through to an advanced membrane filtration system. After two years of operation, the distribution system was shut down and samples of pipe were collected for autopsy analysis. Inlet and outlet samples from each PDS were collected for purpose of comparison. ToF-SIMS was used to assess chemical differences in surface biofilm accumulation and particulate deposition, which resulted as a consequence of the treatment method and operational mode of each system. These data supplemented previously collected bacteriological and chemical water quality data. Results from the inorganic analysis of the pipes were consistent with corrosion and contamination events that occurred upstream in the corresponding treatment systems. Principal component analysis of data on organic constituents showed oxygen and nitrogen containing fragments were associated with the treatment inlet and outlet samples. These types of signals can often be ascribed to biofilm polysaccharides and proteins. A trend was observed when comparing samples from the same PDS, showing an association of lower molecular weight (MW) organic fragments with the inlet and higher MW organic fragments with the outlet samples.

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#### 1. Introduction

It has become evident over the last decade that the influence of the distribution system is one of the largest uncertainties in drinking water quality at the customer tap. This is partly due to aging and unmanaged infrastructure and partly because of the poorly understood interaction between the distribution system infrastructure and water. Control of the water quality entering the distribution system is a key approach to minimise particulate deposition and biodegradable natural organic matter which is a nutrient source for biofilm growth. A number of analytical methods have been developed to allow assessment of the biostability of the water including the Biofilm Formation Rate (BFR) and Biofilm Formation Potential (BFP) tests (van der Kooij et al., 1995). These have been successfully combined with assimilable organic carbon (AOC) as a measure of organic nutrient availability and adenosine tri-

\* Corresponding author. E-mail address: rolando.fabris@sawater.com.au (R. Fabris). phosphate (ATP) as a measure of biofilm activity to establish guidelines for effective distribution system management, especially in undisinfected supplies (van der Kooij, 2000). More recently, there has been a greater focus on distribution systems as dynamic rather than static infrastructure components (Kerneïs et al., 1995). Recent investigations into the characteristics of particles in distribution systems (Vreeburg et al., 2008) and microbial ecology (Douterelo et al., 2013) have revealed their nature as bioreactors, and propagators of sediment accumulation and release. Despite this, standard water quality analyses such as colour and turbidity are still the predominant mode of monitoring water quality, with bacteriological quality based upon maintaining chlorine residual and minimising heterotrophic plate counts.

A number of distribution system investigations have been reported using pilot facilities to study behaviour in a controlled environment. The most common is the 'TORUS' pilot distribution facility, developed by Thames Water (Holt et al., 1994; Smith et al., 1999; Maier et al., 2000; Boxall and Saul, 2005). A number of other investigations have also examined the effect of water quality, nutrients and flow on the bacteriological stability in pilot distribution







systems (Piriou et al., 1998; Volk and LeChevallier, 1999; Frias et al., 2001; Lehtola et al., 2006). Investigations that have examined parallel pilot distribution systems over long periods are uncommon and best represented by the work of Liu et al. (2013) where 3 treatment streams fed 6 mm ID poly-ethylene (PE) pipe rigs of 24 h detention time, evaluated over a 288-day period, with a focus on microbial growth. Findings included the fact that membrane filtration was superior to ion-exchange treatments for controlling particle accumulation in the PDS through superior removal of particulate loadings and that biofilm development was strongly correlated with nutrient reduction.

Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) is an analytical technique used to image and record organic and inorganic mass spectral data of solid materials. It is a highly sensitive technique that provides chemical information regarding elemental, isotopic and molecular composition from the first one to two monolayers under static conditions (Vickerman, 2001). It involves the analysis of ionised particles that are emitted when the surface is bombarded with an energetic primary ion beam. Emitted particles are accelerated to constant kinetic energy into the timeof-flight chamber, where mass separation is achieved according to mass-to-charge ratio. It is a highly surface sensitive technique, as only the secondary ions generated from the outer 10–20 Å region have enough energy to escape the surface for detection and analysis. Although not a quantitative technique, ToF-SIMS can provide a qualitative surface chemistry analysis and comparison.

Following a two year project to determine the level of treatment required to minimize water quality deterioration within multiple pilot distribution systems, representative pipe sections at the beginning and end of each distribution system were removed and autopsied for a variety of physical and chemical parameters, including surface characterisation by ToF-SIMS to evaluate the impact of input water quality on organic and inorganic pipe deposits, especially components that may cause water quality deterioration.

#### 2. Materials and methods

The source water for the study was River Murray water taken from the Mannum to Adelaide pipeline at the Mt. Pleasant water treatment plant (WTP), located in the Adelaide Hills approximately 60 km from Adelaide. The feed waters for the four distribution systems were product water of increasing quality from four different treatments, as detailed below.

#### 3. Treatment processes

**Stream (S1)** – **Conventional (Conv)** comprised alum coagulation followed by flocculation, sedimentation and dual media (sand/ anthracite) filtration. This process was selected as it represents the most widely applied drinking water treatment process adopted in Australia. Aluminium sulphate (as Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>.18H<sub>2</sub>O) was used as the primary coagulant at dose rates dependent on water quality with pH controlled between 6.2 and 6.5. A cationic poly-acrylamide LT22 (BASF Chemicals, Australia) was dosed as a flocculant aid.

**Stream (S2)** – **MIEX plus coagulation (MIEX/Coag)** consisted of pre-treatment using a magnetic ion-exchange resin (MIEX) for DOC removal coupled with coagulation/sedimentation/filtration treatment as a clarification step for turbidity reduction. The MIEX–DOC<sup>®</sup> removal process implemented at Mt. Pleasant WTP has been described in detail previously by Drikas et al. (2011).

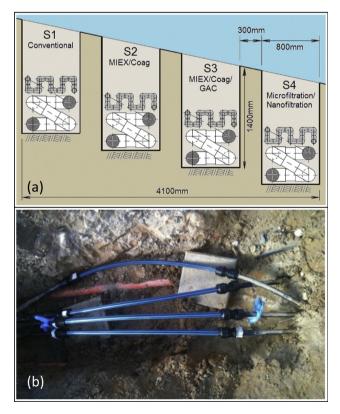
**Stream (S3)** – **MIEX plus coagulation plus GAC (MIEX/Coag/ GAC)** included a further polishing step of granular activated carbon (GAC) after MIEX/Coag (S2). Two gravity fed filter columns filled with F400, a coal based steam-activated GAC (Calgon Corporation, US), were used to achieve an empty bed contact time of approximately 14 min with the product streams combined.

**Stream (S4)** – **Nanofiltration with microfiltration pretreatment (MF/NF)** incorporated dual stage membrane filtration with a Siemens–Memcor submerged microfiltration (MF) pretreatment for particulate removal followed by a DOW–Filmtec NF270 nanofiltration (NF) membrane for DOC removal and hardness reduction. This stream represented an advanced treatment technology and consistently achieved the highest treated water quality. More detailed descriptions of the treatments are available in previous literature (Ho et al., 2012; Fabris et al., 2013).

All treated water streams were disinfected to meet a minimum 'Chlorine contact  $\times$  time' factor – Ct (Baumann and Ludwig, 1962; White, 1975) of 30 mg min/L, according to demand, but deliberately controlled to retain no residual at the inlet to the Pilot Distribution Systems (PDS) following 4 h contact in the treated water storage tank. This strategy was chosen to replicate low-flow ends of distribution systems, where disinfectant residual is often lost and to encourage more rapid establishment of any potential biofilms within the operational duration (26 months). Treated water storage tanks were modified 1000 L HDPE intermediate bulk containers with treated and disinfected water entering through a ported lid and exiting to the distribution systems via a drain valve (separate inlet/outlet).

#### 3.1. Pilot distribution systems

Four independent 1.05 km looped PDS using a combination of 150 mm nominal diameterpolyvinyl chloride (PVC) and 50 mm polyethylene pipe were constructed for this study. Pipes were arranged in 75 m lengths with compact 180° bends allowing inlet and outlet to be collected at the same terminus (Fig. 1a). All pipe work



**Fig. 1.** (a) Pilot distribution system cross-section layout and (b) Replaced 20 mm PE autopsy sections from supply pipes immediately preceding PDS inlet.

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