USING INDICATOR CHEMICALS AND ONLINE SURROGATES TO MANAGE THE CHEMICAL RISK OF RECYCLED WATER

Exploring monitoring strategies for reverse osmosis treatment

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ABSTRACT

The Water Corporation of Western Australia's Groundwater Replenishment Trial (GWRT) has recently tested a new approach for monitoring reverse osmosis (RO) treatment performance using a suite of treatment performance indicator (TPI) chemicals and two online surrogate parameters – conductivity and total organic carbon. It was intended that TPIs would provide a conservative assessment of the removal of unmonitored chemicals with similar physicochemical characteristics. However, while operational and surrogate parameters indicated good RO performance, the calculated removal efficiency of many TPIs failed to meet targets. External factors, including analytical method uncertainty, low feed concentration and, in some cases, the potential for chemicals to form within the treatment process, were found to significantly affect calculated removal.

It is recommended that RO treatment performance monitoring should focus on surrogate parameters that can be measured online, as they provide immediate feedback that can be linked to variation in plant performance. In contrast, a set of chemical indicators chosen to consider health risks posed by a broad array of chemical classes proved valuable in demonstrating the ongoing safety of the recycled water.

Keywords: Chemical risk, groundwater replenishment, reverse osmosis, treatment performance indicator (TPI).

INTRODUCTION

Drinking water augmentation is considered an inherently high-risk use of recycled water because of the public health implications of contaminants in untreated wastewater. While the greatest acute risk to consumers of drinking recycled water remains pathogenic microorganisms (AGWR, 2008; ADWG, 2011), there is also community and scientific concern over chemicals in recycled water. Secondary treated wastewater contains a wide range of household, industrial and agricultural chemicals (Van Buynder *et al.*, 2009; Shon *et al.*, 2006), albeit at low concentrations. Coupled with this, the increasing sensitivity of chemical analysis techniques permits detection of increasing numbers of chemicals in water and wastewater at lower and lower concentrations (e.g., ng/L levels).

Chemicals in drinking water have long been assessed relative to values relevant to human health risk (ADWG, 2011; WHO, 2006; CDPH, 2011; Drewes *et al.*, 2008). However, since 2004, the *Australian Drinking Water Guidelines* (ADWG) have recommended a risk-based management framework utilising multiple barriers, rather than relying solely on compliance of product water with guideline concentrations (ADWG, 2011). This approach was also adopted for the *Australian Guidelines for Water Recycling* (AGWR, 2008), where the focus is on targeted operational monitoring, after an initial validation of chemical and microbiological removal by each treatment process. One approach that has been proposed as a cost-effective means of monitoring treatment processes is the use of treatment performance indicators (TPIs). These are chemicals that provide a conservative assessment of the removal of unmonitored chemicals with similar physicochemical characteristics in a given treatment process (Drewes *et al.*, 2008; AGWR, 2008; Dickenson *et al.*, 2011; Rodriguez *et al.*, 2012). Treatment performance can also be monitored using online surrogates. These are parameters for which quantifiable change in real time can serve as a performance measure of chemical removal.

Reverse osmosis is a key barrier in the removal of chemicals in many water recycling schemes. Chemical rejection by RO is influenced by compound-specific properties (e.g. molecular size, solubility, diffusivity, polarity, hydrophobicity and charge), membrane properties (e.g. permeability, pore size, hydrophobicity and charge), as well as membrane operating conditions (e.g. flux, trans-membrane pressure and membrane-cleaning regime). Based on a comprehensive literature review, Bellona *et al.* (2004) proposed a schema for qualitative prediction of rejection of organic micropollutants (Figure 1), which considers molecular weight (MW), molecular width (MWd), the acid dissociation constant (pKa, which determines whether a molecule will be charged at a given pH), and the octanol-water partition coefficient (Kow, which is a measure of the polarity of a molecule). Use of this schema illustrates that rejection efficiency can be predicted for any chemical based on its physical and chemical properties. Thus it was hypothesised that monitoring a set of chemicals that represent a broad range of physico-chemical properties, in particular size, charge and hydrophobicity, would give confidence that the chemical indicators chosen also account for unknown and new chemicals (Van Buynder *et al.*, 2009).

The Western Australian Groundwater Replenishment Trial (GWRT) was undertaken by the Water Corporation of Western Australia (January 2010 to December 2012) to demonstrate the technical feasibility of groundwater replenishment with recycled water to deliver safe and reliable water (WCWA, 2013). The purpose-built Beenyup Advanced Water Recycling Plant (AWRP) produced up to 5 ML/day of recycled water from secondary treated wastewater that would otherwise be

BOX 1. DEFINITION OF SURROGATE AND INDICATOR CHEMICALS TESTED DURING GWRT

Surrogate A bulk parameter, such as conductivity or total organic carbon, in which a quantifiable change can serve as a performance measure of chemical removal by individual treatment processes. For operational control, surrogates need to be able to be measured and reported in real time.

Treatment Performance Indicator (TPI) An individual chemical, occurring at quantifiable level in source water, that can represent a group of chemicals with similar physicochemical characteristics, relevant to treatment removal. Thus monitoring TPI removal provides a conservative assessment of the removal of those unmonitored chemicals with similar physicochemical characteristics. The criteria for selection of a TPI in this study were:

- Quantifiable using an established and preferably accredited analytical method;
- Frequently detected in feedwater (>80% & preferably at 100% detection);
- The ratio of the concentration of the chemical in the RO feedwater to the limit of reporting (LOR) of the analytical method was greater than five.

Recycled Water Quality Indicator (RWQI) An individual chemical that demonstrates the safety of recycled water for a specific chemical group, providing additional confidence beyond TPI that all chemical hazards are being mitigated. This is particularly useful for chemical classes for which TPIs could not be identified, such as hormones and pesticides. RWQIs were chosen based on the following criteria for selection:

- Quantifiable using an established and preferably accredited analytical method;
- Frequency and concentration of detection in feedwater.

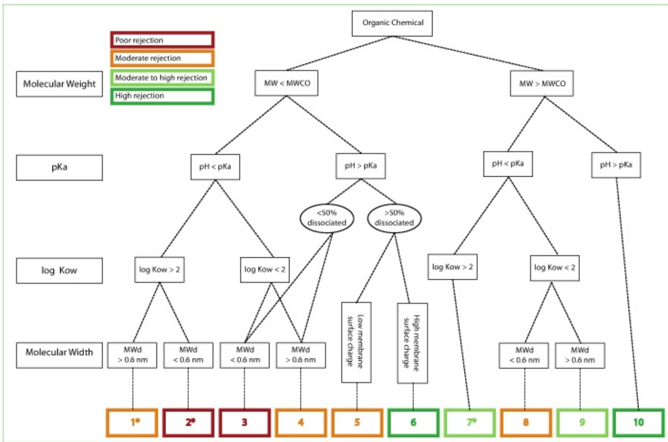

Figure 1. Rejection diagram for organic micropollutants, adapted from Bellona *et al.* (2004) and printed with permission from Elsevier. **Rejection of hydrophobic classes (1, 2, 7) were also impacted by chemical partitioning and diffusion through the membrane.*

discharged to the ocean from Beenyup Wastewater Treatment Plant (WWTP). Wastewater at Beenyup WWTP is treated by a nitrification-denitrification activated sludge process, and then undergoes advanced treatment via ultrafiltration, RO and UV disinfection at the AWRP. The key chemical removal barrier is RO. As well as regular analysis of an extensive suite of 292 recycled water quality parameters in the treated water, TPI chemicals were identified to monitor chemical removal by RO. In addition, the concept of Recycled Water Quality Indicators (RWQIs) was also developed during baseline monitoring (Van Buynder *et al.*, 2009), and trialled during the GWRT to demonstrate the safety of recycled water with respect to specific chemical groups of health concern (see Box 1 for definition of terms).

To our knowledge, GWRT was the first full-scale test of an indicator chemical approach for monitoring RO treatment performance. During the trial, it became apparent that the calculated removals of TPIs through RO were not consistently achieving targets, even though 100% of the water quality samples met health and environmental guidelines. This paper provides an assessment of the TPI data collected during the GWRT, comments on RWQI selection, and recommendations on performance monitoring for recycled water plants utilising RO.

METHODS

Before commencement of the GWRT, baseline monitoring was undertaken at two RO plants, the Kwinana Water Recycling Plant and Beenyup Pilot Plant over three years (2006–2008) to assess the chemical risk associated with wastewater treated through RO, with 396 chemicals assessed before and after RO treatment in 15 chemical classes (Van Buynder *et al.*, 2009; Linge *et al.*, 2010; Linge *et al.*, 2012).

This original research confirmed that online monitoring of total organic carbon (TOC) and conductivity were suitable surrogates to monitor RO treatment and that they were suitable critical control points for the AWRP, and also identified target chemicals to act as TPIs in the trial (Table 1). While it was initially intended that a TPI would be chosen for each of the 15 chemical classes tested, it became apparent that for some chemical classes there were no chemicals detected with sufficient frequency in secondary wastewater (>80% and preferably at 100% detection) to be considered as a TPI (see Table 1).

Given that some of these classes posed a particular health risk (e.g. hormones), a second class of indicator chemical was also developed – the Recycled Water Quality Indicator (RWQI) – to demonstrate the safety of recycled water for a specific chemical group. The criteria for selection of TPI and RWQI are presented in Box 1.

For those chemical classes for which a TPI had been identified, this chemical also acted as the RWQI for that class. While TPI and RWQI selection was based on the percentage detection and concentration of chemicals in secondary wastewater, the

Table 1. Rejection class and target removal efficiency of TPI, shaded in green, plus additional RWQI selected during commissioning of the Beenyup AWRP.

Indicator (chemical group)	MW	RO rejection class, assuming MWCO = 200 Da and pH 7	TPI target removal efficiency	Percentage detection in secondary wastewater (Van Buynder *et al.*, 2009)	Percentage detection in RO treated water (Linge *et al.*, 2012)
Boron (Metals & metalloids)	10.8	3, 5 or 6 depending on membrane surface charge and per cent dissociation	15%	100%	89%
Nitrate (Inorganic anions)	62	5 or 6 depending on membrane surface charge	80%	100%	
NDMA (*N*-Nitrosamines)	74	3		96%	93%
Chlorate (Inorganic anions)	83	5 or 6 depending on membrane surface charge		37%	46%
1,4-Dioxane (Miscellaneous)	88	3	75%	100%	29%
Chloroform (Halogenated disinfection by-products)	119	3		85%	56%
1,4-Dichlorobenzene (VOCs)	147	1 or 2 depending on molecular width		95%	89%
Fluorene (PAHs)	166	1or 2 depending on molecular width		64%	19%
2,4,6-Trichlorophenol (Phenols)	197	3, 5, or 6, depending on membrane surface change and percent dissociation		64%	0%
Carbamazepine (Neutral pharmaceuticals)	236	7	90%	97%	0%
Estrone (Hormones)	270	7		48%	0%
EDTA (Complexing agents)	292	10	90%	100%	48%
Diclofenac (Acidic pharmaceuticals)	296	7	80%	100%	0%
Trifluralin (Pesticides)	335	7		91%	0%
Octachlorodibenzodioxin (Dioxins, furans and dioxin-like PCBs)	460	7		67%	17%

indicators chosen represent a wide range of chemical types, with particular focus on chemicals that are likely to be poorly rejected (e.g. Class 2 or 3). The majority of larger molecules (e.g. MW > MWCO of the RO membrane) chosen are charge neutral and hydrophobic and, therefore, may pass through an RO membrane by adsorption and diffusion processes.

Removal targets for each TPI were determined during commissioning based on observed removal. Treatment performance across RO was calculated using TPI concentrations in samples of ultrafiltration treated water (filtrate) and RO permeate, as per Equation 1. For those parameters not detected in RO permeate, the TPI removal efficiency was calculated assuming an RO permeate concentration equal to the limit of reporting (LOR) of the analytical method. Hence, in these cases the removal efficiency is a conservative estimate.

$$\%\text{Removal} = \frac{[\text{UF Filtrate}] - [\text{RO Permeate}]}{[\text{UF Filtrate}]} \times 100 \quad (1)$$

Factors affecting calculated TPI removal efficiencies were determined using GWRT monitoring data from March 2010 to October 2011. Additionally, RO membrane replacement in December 2011, motivated by deteriorating operational parameters, offered a unique unplanned experiment to evaluate calculated TPI removal efficiencies before and after membrane replacement.

DISCUSSION

FACTORS AFFECTING CALCULATED TPI REMOVAL EFFICIENCY

At face value, if a calculated TPI removal efficiency does not meet a removal target, it suggests that RO is not consistently removing that TPI from the recycled water stream. However, analysis of the GWRT dataset indicated that calculated TPI removal efficiencies were impacted by several external factors, including poor data confidence, the potential for chemicals to form within the treatment process, and low feedwater concentration. In these cases, failure of a TPI to meet a removal target cannot be used as evidence that the RO treatment is not operating properly.

Chemical classes such as disinfection by-products and volatile organic compounds may increase in concentration within MF/RO plants (Linge *et al.*, 2013; Van Buynder *et al.*, 2009; Rodriguez *et al.*, 2012). For disinfection by-products, this increase in concentration is attributed to formation after chloramination, which is used to reduce RO membrane biofouling. Disinfection by-products formation in MF/RO plants can lead to underestimation of removal by RO (Linge *et al.*, 2013; Van Buynder *et al.*, 2009). Increases in volatile organic compound concentrations have been attributed to trace contamination from atmospheric sources (Rodriguez *et al.*, 2012). In this study, 1,4-dichlorobenzene was also frequently detected in both field and trip blanks, potentially influencing some RO removal calculations by up to 25%. Trace contamination may come from sampling equipment, sample preparation, or atmospheric sources.

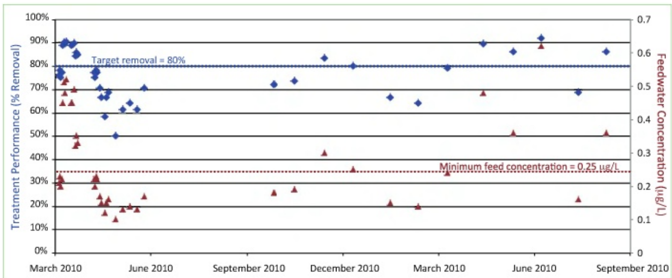

Figure 2. Comparison of calculated treatment performance (blue) and feedwater concentrations (red) for diclofenac.

The impact of data confidence can be illustrated by considering the variability in calculated removal efficiency for each TPI, reported as percentage relative standard deviation (Table 2). Only samples where minimum feed concentrations were achieved were chosen. Chloroform, NDMA and 1,4-dichlorobenzene all show variability in calculated RO rejection that is greater than 100%, and all of these chemicals are likely to be impacted by formation, blank contamination in field and trip blanks, or analytical uncertainty.

Previous calculation of measurement uncertainty for all TPI, except nitrate, showed that the TPI with the highest relative uncertainty is NDMA (40% relative standard deviation). This is the only TPI measured at ng/L concentrations (Van Buynder *et al.*, 2009). Given this large uncertainty, and the low concentrations of NDMA measured in the RO product water (typically <10 ng/L), we estimate the uncertainty for NDMA removal efficiency will be at least 80% relative standard deviation. Even in the absence of any other form of uncertainty (e.g. from sampling or contamination), this large method uncertainty has a significant impact on the reliability of NDMA as a TPI candidate.

Finally, removal percentage is fundamentally limited as a performance metric for TPI, as accurate calculation requires the TPI to always be detected in the feedwater, and never occur below detection levels in the RO permeate. When a TPI is not detected in RO permeate, the calculated removal will be influenced by the LOR of the analytical method and calculated removal is underestimated. This effect is demonstrated for diclofenac (Figure 2), which was never detected in RO permeate and therefore calculation of removal efficiency always used the LOR value (0.05 µg/L). Target removal of 80% for diclofenac could only be demonstrated when feedwater concentrations were above a 'minimum feed concentration' of 0.25 µg/L and, therefore, calculated removal could not be used to monitor RO performance when feedwater concentrations were below 0.25 µg/L.

Similar patterns were seen for EDTA and 1,4-dioxane, indicating that the reason that these compounds show calculated removal below target was because feed concentrations were too low and not due to RO underperformance. In contrast, carbamazepine concentrations were always above the minimum required in feedwater, and calculated removal always met the target. Furthermore, once the analytical method for EDTA was modified such that the LOR reduced from 10 to 1 µg/L, both minimum feed concentration and target removal were always achieved, despite EDTA never being detected in RO permeate.

Table 2 summarises the impact of the factors described on the calculated removal for each TPI. Only two TPI, carbamazepine and nitrate, were not affected by either variable feed concentration or poor data confidence. However, carbamazepine is a large molecule and is unlikely to be sensitive to changes in RO removal efficiency (Bellona *et al.*, 2004). Nitrate is more likely to be sensitive to changes in RO, although it is still expected to have moderate membrane rejection (i.e. removal) because of electrostatic repulsion.

SENSITIVITY OF TPI RESPONSE COMPARED TO CONDUCTIVITY

The intended use of TPIs and surrogate parameters during the GWRT was to indicate changes in performance in RO treatment, such that *'If the level of removal of the indicator compound is significantly diminished, it will indicate reduced system performance'* (Van Buynder *et al.*, 2009). However, analysis of GWRT data demonstrated that no TPI showed a clear reduction in RO removal with membrane age, due in part to the factors discussed above.

In December 2011, the RO membranes in the AWRP were replaced because of decreased operational performance (e.g. excessive trans-membrane pressure drop and high electrical conductivity of the permeate). Figure 3 shows that the conductivity in RO permeate significantly decreased on membrane changeout, while the RO conductivity removal efficiency, calculated using online measurements that matched TPI sampling times, increased. This confirmed that conductivity was sufficiently sensitive to reflect dramatic changes in RO performance, such as when membranes are replaced.

In contrast, the calculated removal efficiencies for large molecular-sized TPIs from Class 7 or 10 (e.g. carbamazepine, diclofenac, EDTA) showed no difference after RO changeout (data not shown). Figure 3 also shows that calculated removal efficiencies for NDMA remained variable both before and after RO changeout, and this was similar for other 'sensitive' TPIs with poor data confidence (e.g. chloroform, 1,4-dichlorobenzene, 1,4-dioxane and boron). The only TPI (Figure 3) for which calculated removal efficiency improved after RO changeout was nitrate (89 ± 2.2 after, compared to 84 ± 3.1 before). This finding is in agreement with our previous conclusion that nitrate was the only 'sensitive' TPI with good data confidence.

PRACTICAL CONSIDERATIONS FOR TPI ANALYSIS

While it is certainly more cost effective to focus chemical analysis on a select list of 10 or 20 chemicals, rather than hundreds, trace chemical analysis at µg/L or ng/L concentrations is still time consuming and expensive. Laboratory turn-around times for off-line measurement of TPI ranged from 12–25 working days (WCWA, 2012). Even if nitrate were a suitable TPI, its current turnaround time of 14 days means it could not be used to indicate a deterioration in RO removal in a timely manner. Operational monitoring must be conducted in real time to enable quick response to changes. Monitoring of critical control points at the GWRT is already undertaken using a number of online parameters, including conductivity, TOC and turbidity.

Table 2. Frequency that target removal efficiency is achieved compared to the effect of variable feed concentration and data confidence on calculated target removal for each TPI.

TPI Chemical	Frequency target removal efficiency is achieved	Frequency that feed concentration was sufficient to demonstrate target removal	Variability in calculated removal (% relative standard deviation)	Suitability as a treatment performance indicator
Boron	90%	100%	34.9%	Method uncertainty impacts calculated RO removal, but potentially sensitive TPI
Nitrate as N	98%	100%	3.9%	Moderately sensitive TPI
NDMA	50%	97%	157%	Large analytical uncertainty and potential for formation in AWRP
1,4-dioxane	88%	88%	2.7%	Insufficient feed concentration
Chloroform	53%	100%	137%	Moderate measurement uncertainty and clear evidence of formation in AWRP
1,4-dichlorobenzene	70%	100%	159%	Moderate measurement uncertainty and frequent contamination in blanks
Carbamazepine	100%	100%	1.0%	High data confidence but insensitive TPI
EDTA	52%	52%	2.2%	Target removal achieved 100% of time once LOR was reduced from 10 µg/L to 1 µg/L. But insensitive TPI
Diclofenac	40%	40%	2.9%	Insufficient feed concentration

Online measurements of specific chemicals, such as colorimetric analysis of nitrate, sulfate, chlorine, ammonia and monochloramine, may provide a reliable measurement of sensitive indicators in addition to surrogate measurements. However, more research is required to determine if this would be appropriate in practice. Online analysis may also overcome issues of trace contamination from sampling equipment and sample preparation.

RECYCLED WATER QUALITY INDICATORS (RWQI)

The USEPA's most recently published strategy for drinking water standards (US EPA, 2010) now contains the key principle that contaminants should be addressed as a group for cost-effective drinking water quality, and that new treatment technologies should address health risks posed by a broad array of chemicals. A recent Recycled Water Science Advisory Panel review convened by the California State Water Resources Control Board also recommended monitoring of both health-based and performance-based indicators for chemicals of emerging concern in recycled water (Anderson *et al.*, 2010).

An Australian study, which evaluated bioanalytical tools for determining recycled water safety, has also suggested a tiered structure of chemical risk management, in which prioritisation of health indicators is based on relevance and feasibility of *in vitro* testing systems (Chapman *et al.*, 2011). The analysis of 15 RWQIs during the GWRT, in conjunction with an extensive suite of 292 recycled water quality parameters, is one of the first explicit tests of the concept that a group of chemicals can be represented by analysis of a single chemical (RWQI), rather than analysis of every chemical in that group. The RWQIs chosen in this study confirmed the safety of recycled water at the GWRT; however, the occurrence pattern of trace organic chemicals in treated wastewater is country-specific (Dickenson *et al.*, 2011). Hence, any proposed indicator requires an occurrence survey to provide data on detection frequency and concentration before it can be adopted.

CONCLUSIONS

In practice, TPIs were not able to demonstrate RO membrane performance as anticipated. Analysis of GWRT data indicated that calculated TPI removal efficiency was affected by many external factors (low concentration in feedwater, formation during water treatment and atmospheric contamination), and the results for all TPIs *except* carbamazepine, EDTA and nitrate were affected in this way. Prior to a membrane changeout in December 2011, there was no measureable decline in calculated TPI removal efficiency, and the only TPI sufficiently sensitive to respond to membrane changeout was nitrate. Surrogates (i.e. conductivity) and operational parameters were better indicators of changes in RO performance than the TPIs, and it is recommended that RO treatment performance monitoring should focus on online surrogates and operational parameters, as they provide an immediate response that can be linked to variation in plant performance effectively in real time. Furthermore, they are easily available as online instruments, and are typically implemented in automated control systems.

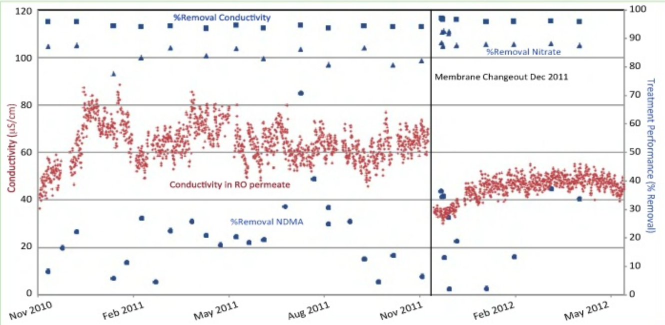

Figure 3. Effect of membrane changeout on conductivity measured in RO permeate, and the calculated removal efficiency of conductivity, nitrate and NDMA.

While TPIs were not able to sufficiently monitor RO treatment performance, the monitoring undertaken during the GWRT generated an extremely valuable dataset, which led to a greater understanding of how the AWRP operates. In addition, RWQIs demonstrated the safety of recycled water at the GWRT, and chemical indicators based on health considerations are gaining traction internationally. While guidance for appropriate health-based indicators can be found in the literature, the occurrence pattern of trace organic chemicals in treated wastewater is location-specific. Thus chemical indicators must be chosen using sound evidence, including assessment of feedwater concentrations, RO removal and a clear understanding of their health risk, before adoption.

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