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# Highly treated mine waters may require major ion addition before environmental release

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#### HIGHLIGHTS

- ► A treated waste water product was assessed using ecotoxicology and TIE methods.
- ▶ Water quality of the distillate was markedly improved but residual toxicity remained.
- ▶ Major ion deficiency was identified as the primary cause of effects to Hydra sp.
- ▶ Residual metal was measured at concentrations that may have contributed to toxicity.
- Treated waters may have inadvertent environmental effects that need to be managed.

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#### ABSTRACT

Mining operations often use passive and/or active water treatments to improve water quality prior to environmental release. Key considerations in choosing a treatment process include the extent to which the water quality is actually improved, and the potential residual environmental risks of the release of such water. However, there are few published studies concerning the environmental impacts of treated waste waters. This study used toxicity identification evaluation (TIE) methods to quantify and identify the "toxic" constituents of a highly-treated water (distillate) produced by brine concentration of a mining process water. Exposure of five freshwater species (Chlorella sp., Lemna aequinoctialis, Hydra viridissima, Moinodaphnia macleayi and Mogurnda mogurnda) to a concentration range of the distillate (0, 25, 50 and 100%) found that it was toxic to H. viridissima (50-100% effect when exposed to 100% distillate). TIE tests demonstrated that the effect wasn't due to residual ammonia ( $\sim 1 \text{ mg L}^{-1} \text{ N}$ ) or trace organics, and unlikely to be due to manganese (Mn; 130–230  $\mu$ g L<sup>-1</sup>). Conversely, addition of 0.2 and 0.5 mg L<sup>-1</sup> calcium improved the growth rate of *H. viridissima* by 61 and 66%, respectively, while addition of calcium, sodium and potassium (0.5, 1.0 and 0.4 mg  $L^{-1}$ , respectively) to levels comparable to that in the local aquatic environment resulted in 100% recovery. Further assessment on the likelihood of residual metal toxicity indicated that Mn concentrations in the distillate were at levels that could inhibit the growth of *H. viridissima*. Ultimately, the results demonstrated that ion deficiency should be considered as a potential stressor in risk/impact assessments of the discharge of treated wastewaters, and these may need to be supplemented with the deficient ions to reduce environmental impacts. The findings have highlighted the need for water managers to consider the possibility of unintended environmental risks from the discharge of highly-treated wastewaters.

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#### 1. Introduction

Depending on the resource being exploited, mining operations can produce gigalitres of saline, acidic, turbid and/or metal contaminated mine and process waters (Lottermoser, 2010). These waters represent a major economic and potential environmental liability and need to be managed appropriately (Mudd, 2007). Hence, there are numerous examples of mining operations that use passive and/or active water treatment methods to improve water quality prior to environmental

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release (Allen, 2008; Banks et al., 1997; Butler et al., 2011; Driussi and Jansz, 2006; Masarczyk et al., 1989). Some costly active treatment processes, such as reverse osmosis and distillation, are capable of producing high-purity waters that contain constituents that are near or below analytical detection limits and have very low electrical conductivity (EC) (Lottermoser, 2010). However, where such highly-treated waters are discharged to the environment, it should not be assumed that they will be environment may still exist due to residual toxicity caused by toxicants that were not effectively removed by the process, and/or a lack of essential ions. For example, a reverse osmosis (RO) treated sewage water was determined to pose an unacceptable environmental risk,

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due to ion deficiencies, if the EC of the water was  $<120 \ \mu$ S cm<sup>-1</sup> (Griffith and Biddulph, 2010). Consequently, waste water managers need to consider unintended risks such as those associated with ion deficiency.

Key considerations in choosing an appropriate treatment process include the intended end use of the treated water, the extent to which the treatment process improves water quality, and the residual risks of the use or release to the environment of such water. The residual (or acquired) toxicity of a treated water can be assessed using traditional ecotoxicological protocols, while toxicity identification evaluation (TIE) may be able to identify the toxic constituents of the water (Mirenda and Hall, 1992). Toxicity identification evaluations involve specific manipulations (e.g. pH adjustment, EDTA additions, sodium thiosulfate additions, and C18 solid phase extraction) of a whole effluent in order to change the amount and/or speciation/bioavailability of potential toxic constituents. The subsequent level of toxicity of the manipulated water relative to the unmanipulated water provides information on the likely toxic constituents. TIE methods have been commonly used to identify toxic constituents of treated sewage (Adams et al., 2008; Bailey et al., 2000) but, to our knowledge, they have never been used to assess the toxicity of a highly-treated mine water product.

The present study assessed, and identified the causes of, the residual/ acquired toxicity of a distillate produced from brine concentration (also referred to as falling film evaporation) of process water sourced from the tailings storage facility at the Ranger uranium mine. The mine is surrounded by Kakadu National Park, which has a World Heritage listing for both its natural and cultural values, and Ramsar listing for its wetlands of international importance.

Brine concentration involves the cascading of heated process water down a falling film tube bundle, with the vaporised fraction then being compressed to heat the falling film tubes and finally condensed into a purified distillate product. The objectives of the study were to: (i) detect and quantify any residual toxicity of the distillate and, (ii) in the event effects were observed, to identify the toxic constituent(s) of the distillate using TIE methods.

#### 2. Methods

#### 2.1. General laboratory procedures

All plastics and glassware were washed by soaking in 5% (v/v) nitric acid for 24 h before being washed with a non-phosphate detergent (Gallay Clean A powder, Gallay Scientific, Burwood, Australia) in a laboratory dishwasher operated with reverse osmosis/deionised water (Elix, Millipore, Molshiem, France). All reagents used were of analytical grade and stock solutions were made up in high purity water (18 M $\Omega$ , Milli-Q Element, Millipore, Molshiem, France).

#### 2.2. Test waters

The distillate was produced from Ranger uranium mine process water by a pilot-scale brine concentrator, which used a falling film evaporation process. Two separate batches of the distillate were collected from the brine concentrator for toxicity testing. The first batch was a 20 L composite sample collected from 11 to 17 July 2011, and was used for the initial screening toxicity tests involving three species (see Section 2.4). The second batch was a 20 L grab sample collected on 10 August 2011, and was used for the remainder of the toxicity and TIE tests. This sample was collected as a grab because the pilot plant project was due to be terminated. Both batch samples were collected in acid-washed high-density polyethylene containers and immediately air-freighted at 4 °C to the Environmental Research Institute of the Supervising Scientist laboratory.

On receipt of the samples, the distillate was immediately sub-sampled for physico-chemical analyses. Specifically, pH (SenTix41 probe, WTW, Weilheim, Germany), dissolved oxygen (DO; CellOx 325 probe, WTW), electrical conductivity (EC; TetraCon 325 probe, WTW) and dissolved organic carbon (DOC; TOC-V CSH, Shimadzu, Kyoto, Japan) were measured in-house. Additional sub-samples were analysed by external laboratories for alkalinity (APHA2320B), total and filtered (<0.45  $\mu$ m) metals (inductively coupled plasma-mass spectrometry (ICP-MS) full scan), nitrate, phosphate, ammonia (colourimetric methods, EPA 353.2, EPA 365.1 and EPA 350.1), and volatile and semi-volatile organic compounds (gas chromatography-mass spectrometry (GC-MS) scan).

#### 2.3. Test diluent

Natural Magela Creek water (MCW) obtained from Bowerbird Billabong (latitude 12° 46′ 15″, longitude 133° 02′ 20″) was used as the control treatment and for dilution of the distillate samples in all tests. The water was collected and transported to the laboratory in 20 L acid-washed plastic containers, and stored at  $4\pm1$  °C prior to filtration through a 3.0 µm pore size filter (Sartopure PP2 depth filter MidiCaps, Sartorius, Göttingen, Germany). Filtration of the natural water was required to remove wild organisms that could confound the results of the toxicity tests, e.g. species that prey on the test subjects. Throughout the testing period, the MCW had a pH of 6.2–6.8 units, an EC of 15–20 µS cm<sup>-1</sup> and DO of >90% saturation.

Diluent water was sub-sampled and analysed for the same physicochemical parameters described for the distillate, and a more limited metal and major ion suite (i.e. totals only; Al, Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, Se, U, Zn, Ca, Mg, Na and  $SO_4$  (analysed as S and converted); referred to as the standard metal and major ion suite hereafter).

#### 2.4. Toxicity test species and methods

The toxicity of the distillate was assessed using five Australian tropical freshwater species: the unicellular green alga (Chlorella sp.); the duckweed (Lemna aequinoctialis); the green hydra (Hydra viridissima); the cladoceran (Moinodaphnia macleayi); and the Northern trout gudgeon (Mogurnda mogurnda). All the organisms were originally isolated from the soft surface waters in Kakadu National Park and have been cultured continuously at the Environmental Research Institute of the Supervising Scientist over many years (10-25 years depending on the species). The optimised and standardised protocols for the toxicity tests are described in full in Riethmuller et al. (2003; http://www. environment.gov.au/ssd/publications/ssr/173.html). Key details of each test are provided in Table 1. For the *L. aequinoctialis* and *Chlorella* sp. tests, nutrients (nitrate and phosphate) were added at the minimum concentrations that would sustain acceptable growth (see Table 1). The MCW used in the Chlorella sp. tests also had 1 mM HEPES buffer added to maintain a stable pH.

Initial toxicity screening of the first batch of the distillate was conducted with a limited range of dilutions of the distillate using three species which had previously displayed sensitivity to process water permeate produced by a microfiltration/reverse osmosis treatment process (van Dam et al., 2011). Specifically, *Chlorella* sp. (72-h cell division rate), *H. viridissima* (96-h population growth rate) and *M. macleayi* (3-brood reproduction) were exposed to the MCW control and three dilutions of the distillate in MCW (i.e. 0, 25, 50 and 100% distillate).

The toxicity of the second batch of the distillate was assessed using the same three species used for the first batch of the distillate (*Chlorella* sp., *H. viridissima* and *M. macleayi*), although only at 0 (MCW control) and 100% distillate concentrations, in order to assess the inter-batch reproducibility of the test methods. Further toxicity testing was also conducted on the second batch of the distillate with two different species, *L. aequinoctialis* (96-h growth rate) and *M. mogurnda* (96-h larval survival) using the same concentration range, 0, 25, 50 and 100% distillate.

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