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Environmental fate of amine oxide: Using measured and predicted values to determine aquatic exposure

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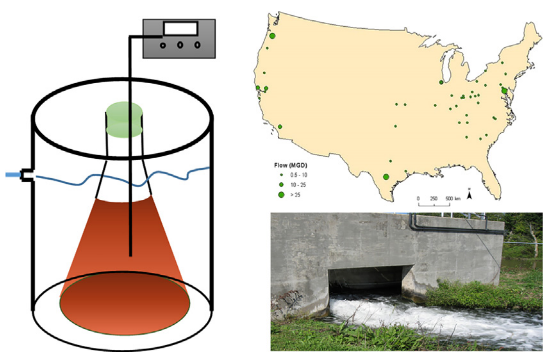
HIGHLIGHTS

- Evaluation of amine oxide fate during down the drain disposal
- Evaluation of biodegradation rate in OECD 314A Sewer Water Die-Away
- Evaluation of fate during Wastewater Treatment Plant Simulation Studies
- Monitoring amine oxide concentrations in US wastewater treatment plant effluents
- Probabilistic exposure modeling to predict concentrations in US receiving streams

GRAPHICAL ABSTRACT



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ABSTRACT

Amine oxide (AO) surfactants are used widely in North American household detergents resulting in > 44,000 mtons disposed down the drain annually. Due to AOs substantial down the drain disposal volume, wide dispersive use, and high aquatic toxicity, there is a need to evaluate ecological exposure and corresponding risk. This study refined the current knowledge regarding the fate of AO disposed down the drain through laboratory simulation studies to evaluate biodegradation in the sewer and during activated sludge wastewater treatment. A monitoring program which measured effluent AO concentrations for the dominant carbon chain lengths, C12 and C14, at 44 wastewater treatment plants (WWTP) across the continental US was also conducted. The study results were then used as input into probabilistic exposure models to predict US receiving stream concentrations. In three separate OECD 314A Sewer Water Die-Away studies AO was rapidly biodegraded with >76% mineralized by study completion and the geometric mean of the primary biodegradation rates being 0.184 h^{-1}. Two OECD 303A Activated Sludge WWTP Simulation studies showed rapid and complete biodegradation of AO with ≤0.09% of parent AO remaining in the effluent, ≤0.03% of parent AO sorbed to sludge solids, and >97% complete mineralization of AO. Monitoring at US WWPTs confirmed low levels of AO in effluents with mean C12 and C14AO concentrations of 52.8 and 20.1 ng/L respectively. Based on the monitoring data, the 90th percentile concentrations of C12 and C14AO for 7Q10 low flow stream conditions were >2 orders of magnitude lower than the predicted no effect concentrations indicating negligible aquatic risk from AO in US receiving streams. This study verifies that AO is safe for the aquatic environment even at the currently high usage volumes due to rapid biodegradation during transit through the sewer and wastewater treatment.

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1. Introduction

Amine oxide (AO) surfactants are widely used in household detergents and hard surface cleaners at active concentrations between 0.1 and 10% (Sanderson et al., 2009). AOs contain even numbered alkyl chains ranging from C8 to C20 and are amphoteric surfactants moving from cationic to zwitterionic to nonionic as pH increases (Singh et al., 2006). The most common AO in commerce is C10-C16 dimethyl AO (CAS# 70592-80-2) with C12 and C14 being the dominant chain lengths accounting for >90% w/w of the mixture and resulting in an average carbon chain length at the point of product formulation in the United Sates (US) of 12.9 (Sanderson et al., 2009).

Because AO is used in consumer detergents and cleaning products which are disposed down the drain, after product use AO will enter the sewer system as a constituent of wastewater (Cowan-Ellsberry et al., 2014). Past research indicates that during transit through the sewer and wastewater treatment plant AO will be biodegraded (Garcia et al., 2007; Sanderson et al., 2009). The biodegradation will likely alter the chain length distribution which changes the ecotoxicity of AO (Belanger et al., 2016). There are no existing simulation studies quantifying AO biodegradation rates in the sewer system; however, Sanderson et al. (2009) used monitoring data to estimate in-sewer loss rates as high as 98% for parent AO. Garcia et al. (2007) found that AO was ready biodegradable under aerobic conditions when exposed to inoculum from an activated sludge wastewater treatment plant. A 2001 monitoring study reported AO (C12 and C14) concentrations in US effluents ranging from 0.4 to 2.91 μg/L (Sanderson et al., 2009).

The aquatic toxicity of AO is well studied and was recently summarized in Belanger et al. (2016). In general AO is highly toxic to aquatic organisms. Recently, Belanger et al. (2016) built on historical AO aquatic toxicity knowledge by developing C8-C16AO acute Quantitative Structure Activity Relationships (QSARs) for a fish (*Danio rerio*), an invertebrate (*Daphnia magna*) and an algae (*Desmodesmus subspicatus*). They also developed a chronic toxicity QSAR for *Desmodesmus* which is the most sensitive taxon. In addition, *Ankistrodesmus falcatus* (algae) and *Lemna gibba* (macrophyte) were studied to increase taxonomic diversity allowing them to generate a 10 Species Sensitivity Distribution (SSD) for AO aquatic toxicity studies. The SSD 5th percentile hazardous concentration (HC5) for C12AO was 52 μg/L.

An environmental risk assessment (ERA) for amine oxides was conducted under the Organization of Economic Cooperation and Development (OECD) high production volume (HPV) chemical program in 2006 and was summarized in Sanderson et al. (2009). A European Union REACH dossier was compiled for AO with study summaries available online by searching the European Chemical Agency's website by chemical name (https://echa.europa.eu/). In all historical assessments AO was shown to be safe at predicted volumes of use due to its rapid biodegradation in the sewer, during wastewater treatment, and in receiving streams. During the HPV assessment in 2006, AOs predicted volume of use in the US was 26,000 metric tons but by 2015 the volume had increased to 44,300 metric tons in North American, NA (Janshekar et al., 2016) (Canada volume of use accounts for 10% of total NA volume therefore the US volume of use has nearly doubled in the past 10 years). Due to AOs continued increase in volume of use, widespread dispersive use, and high toxicity to aquatic organisms, work was undertaken to improve the existing knowledge on AOs fate in the environment and to quantify exposure. This work included laboratory studies aimed at quantifying AOs biodegradation rate in sewer systems and AOs biotransformation and mineralization during wastewater treatment. A monitoring program was also conducted to quantify current levels of AO in WWTP effluents across the continental US.

New approaches and datasets have recently been presented that can be used to conduct probabilistic risk assessments for consumer product ingredients disposed down the drain (Federle et al., 2014; Kapo et al., 2015; Kapo et al., 2017; McDonough et al., 2016; McDonough et al., 2017; Simonich et al., 2013). Kapo et al. (2017) published US distributions of sewer water transit time and Kapo et al. (2015) published US distributions of per capita water use and WWTP receiving stream dilution factors (DFs) under mean and 7Q10 low flow (lowest river flow occurring over a 7-day period every 10 years) conditions. These distributions (sewer transit time, per capita water use, and DFs) can be combined with sewer and wastewater treatment plant simulation studies (which were conducted as part of this research) to create a probabilistic model that predicts exposure concentrations in US WWTP effluents and receiving streams.

Approaches have also been developed which combine distributions of WWTP effluent monitoring data with DFs to predict in-stream concentrations of down the drain disposed chemicals. Federle et al. (2014) presented an approach to predict HHCB (1,3,4,6,7,8-hexahydro-4,6,6,7,8,8-hexamethylcyclopenta-γ-2-benzopyran, trade name Galaxolide) concentrations in US WWTP mixing zones by combining a distribution of measured effluent HHCB concentrations with a distribution of mean or 7Q10 low flow DFs. McDonough et al. (2017) used this approach to predict mixing zone concentrations of OTNE (1-(1,2,3,4,5,6,7,8-octahydro-2,3,8,8-tetramethyl-2-naphthyl)ethan-1-one; trade name Iso E Super) based on US WWTP effluent concentrations. And in another study McDonough et al. (2016) built on this approach and combined distributions of measured effluent anionic surfactant concentrations (alcohol sulfates, alcohol ethoxysulfates, linear alkyl benzenesulfonates, and methyl ester sulfonates) normalized to corresponding effects data and distribution of mean or 7Q10 low flow DFs to evaluate the aquatic risk for each surfactant and the class of anionic surfactants. The current study leveraged these approaches and used effluent monitoring data to predict in-stream AO concentrations and evaluate aquatic risk.

The first objective of this research was to conduct laboratory simulation studies to (1) generate the rate of primary and ultimate biodegradation of AO in the sewer and (2) to quantify the levels of parent AO and metabolites in effluent following activated sludge wastewater treatment. The second objective was to conduct a robust monitoring program to quantify the levels of C12 and C14AO in US WWTP effluents. The final objective was to develop probabilistic models that utilize the laboratory simulation study results (sewer and wastewater treatment plant) and monitoring data as input parameters to predict AO concentrations in US WWTP mixing zones and to quantify ecological risk.

2. Materials and methods

2.1. Overview

This manuscript reports results from laboratory studies, field monitoring and probabilistic modeling therefore the supplementary data has been utilized extensively to provide as much detail as possible regarding each study.

2.2. Laboratory studies

2.2.1. OECD 314A sewer water die-away

1-[^{14}C]-Dodecyldimethylamine oxide (^{14}C12AO) was used in all laboratory studies because it is the predominant chain length of AO disposed of down the drain (Sanderson et al., 2009). Since AO is already used in commerce and therefore present in the collected wastewater at appropriate environmental levels, for both the OECD 314A and 303A studies AO was dosed at trace levels to obtain as accurate kinetic and biodegradation data as possible. Three OECD 314A Sewer Water Die-Away Studies (OECD, 2008) were conducted following the test guidelines. Briefly, ^{14}C12AO was dosed into wastewater from two different WWTPs (Fairfield and Mason OH) and held at 15 °C and 0.5 mg/L dissolved oxygen (DO) to simulate sewer conditions. Liquid scintillation counting (LSC) and radiolabeled thin layer chromatography (Rad-TLC) were used to quantify parent and metabolite levels over the course of the study. Extracted sludge solids were combusted to quantify the

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