Environment International 120 (2018) 172–180

Contents lists available at ScienceDirect

Environment International

journal homepage: www.elsevier.com/locate/envint

Population histamine burden assessed using wastewater-based epidemiology: The association of 1,4-methylimidazole acetic acid and fexofenadine

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ARTICLE INFO

Handling Editor: Yong Guan Zhu

Keywords:
Wastewater-based epidemiology
Histamine
Allergy
Antihistamine
Population health
LC–MS/MS

ABSTRACT

Systematic sampling and analysis of wastewater has become an important tool for monitoring consumption of drugs and other substances, and has been proposed as a method to evaluate aspects of population health using endogenous biomarkers. 1,4-methylimidazoleacetic acid (MIAA) is an endogenous biomarker and metabolite of histamine turnover. Its urinary excretion is elevated in conditions such as mastocytosis, hay fever, hives, food allergies and anaphylaxis. The aim of this study was to develop and apply methods for MIAA in wastewater and compare its occurrence with antihistamine use in wastewater. Consecutive daily samples were collected from seven catchments serving populations from 3000 to 2 million and covering rural and urban communities during the 2016 Census in Australia. MIAA and the antihistamines (ranitidine, fexofenadine, cetirizine) were quantified consistently. Per capita excretion of MIAA (mg/d/capita) estimated from the WW concentrations were consistent with findings from previous clinical studies. We found significant positive correlations between loads of MIAA and fexofenadine ($R^2 = 0.68$, $p < 0.0001$) and cetirizine ($R^2 = 0.25$, $p = 0.03$) across the various catchments. Sewer reactor experiments on the degradation of MIAA and the antihistamines found that fexofenadine is stable for at least 24 h while MIAA, ranitidine and cetirizine are subject to degradation, and this should be considered in interpretations. To the best of our knowledge, this study is the first wastewater study to introduce and monitor an endogenous metabolite of histamine, and the first study to monitor and relate proxies of disease and treatment of disease.

1. Introduction

1.1. Histamine and inflammation

Allergic diseases are prevalent in developed nations, and are becoming increasingly common in developing nations as they increasingly adapt Western lifestyles (Pawankar et al., 2013). For example, the World Allergy Organisation found allergic rhinitis affects 10–30% of adults (Pawankar et al., 2013). While less prevalent (0.5–1% of population), urticaria predisposes to depression, anxiety and sleep difficulties (Balp et al., 2015; Maurer et al., 2011). Mastocytosis is a rare (prevalence: 0.1% of population) but underdiagnosed, multifaceted and sometimes fatal disease (Siebenhaar et al., 2014).

Histamine is a vasodilator that mediates allergic and inflammatory reactions in humans (Xie and He, 2005). Most physiological histamine is stored in mast cells and basophils which, upon activation through immunoglobulin E cross-linkage, release histamine and other cytokines (Taylor et al., 1990). Many of the symptoms associated with allergies (flushing, itch, nasal congestion, headache etc.) occur through the activation of the histamine H1 receptor (HRH1) by histamine (Simons, 2004). Histamine and histamine turnover is subsequently a common denominator of diseases such as mastocytosis (abnormal mast cell proliferation), as well as many allergic diseases such as allergic rhinitis (hay fever), asthma, urticaria (hives), food allergies and anaphylaxis (Gould and Sutton, 2008; Simons, 2004).

Symptoms of allergic rhinitis, urticaria and mastocytosis are treated

Abbreviations: MIAA, 1,4-methylimidazole acetic acid; WBE, Wastewater-based epidemiology; HRH1, histamine H1 receptor; COD, chemical oxygen demand; SCOD, soluble chemical oxygen demand; HRT, hydraulic retention time; RM, rising main; GS, gravity sewer; CS, control sewer

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https://doi.org/10.1016/j.envint.2018.08.009
Received 29 June 2018; Received in revised form 2 August 2018; Accepted 3 August 2018
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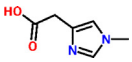
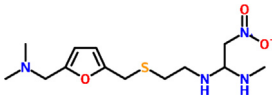
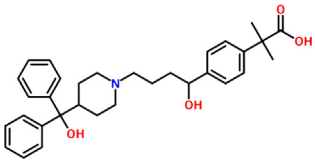
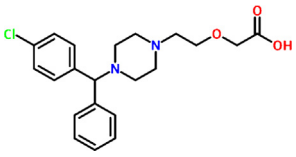
Table 1
Biomarkers featured in this study.

Biomarker	Category	Chemical structure	DDD (mg)	Urinary excretion after oral dose
MIAA	Endogenous histamine metabolite		N/A	N/A
Ranitidine	Antihistamine (H2 histamine receptor agonist)		300	30% of a 150 mg dose (Lauritsen et al., 1990)
Fexofenadine	Antihistamine (histamine H1 receptor agonist)		120	11% of a 120 mg dose (Mikiko et al., 2006)
Cetirizine	Antihistamine (histamine H1 receptor agonist)		10	60% of a 10 mg dose (Spencer et al., 1993)

DDD: Daily defined dose, retrieved from https://www.whocc.no/atc_ddd_index/ (Accessed 22 May 2018). N/A: not applicable.

with antihistamines (Brockow and Ring, 2011; Siebenhaar et al., 2014; Simons and Simons, 2011). H1 antihistamines such as fexofenadine and cetirizine inhibit activation of HRH1 by histamine to quell allergic symptoms. Conversely, H2 histamine receptors (HRH2) which have a limited role in allergic symptoms, are targeted by H2 antihistamines such as ranitidine (Simons and Simons, 2011; Thurmond et al., 2008). Ranitidine inhibits HRH2 expressed by parietal cells of the stomach to decrease stomach acid production, and is used to treat gastroesophageal reflux disease (acid reflux (Dave et al., 2004; Xie and He, 2005)). All three of these antihistamines are excreted in urine after administration (Table 1). Therefore, short-term consumption of these antihistamines can be detected through urinalysis.

N-tau methylmidazoleacetic acid (MIAA) (Table 1) is the main urinary metabolite of histamine (Schayer and Cooper, 1956; Tham, 1966). MIAA is an indicator of histamine turnover and mastocytosis (Doormaal et al., 2012; Martens-Lobenhoffer and Neumann, 1999; van der Veer et al., 2012). Its excretion also reflects blood histamine levels to a limited degree (Taylor et al., 1990), and is increased during mastocytosis, urticaria, atopic dermatitis, allergic rhinitis and other conditions involving high histamine turnover (Doormaal et al., 2012; Holm-Bentzen et al., 1987; Ib, 1982; Maintz and Novak, 2007; Mallet et al., 1989). A more extensive summary of conditions implicating MIAA is provided in Table A.1. Urinary MIAA can therefore provide a non-invasive insight into the histamine burden of individuals (Doormaal et al., 2012; Maintz and Novak, 2007).

1.2. Wastewater-based epidemiology and population health

Municipal wastewater is akin to a pooled population urine sample; it is a dynamic depository of countless chemicals that reflect upon individuals' interactions with various chemical and environmental factors. Therefore, aspects of population health can be deduced by measuring biomarkers of known cause or origin. This is the basis of wastewater-based epidemiology (WBE), a technique commonly used to measure population consumption of licit drugs (Rodríguez-Álvarez et al., 2015) illicit drugs (van Nuijs et al., 2011) and personal care products (Kasprzyk-Hordern et al., 2008). Numerous pharmaceuticals, including antihistamines have also been measured with WBE to infer about population health (Gracia-Lor et al., 2017). For example, the antihistamine cetirizine has been shown to correlate with seasonal pollen levels (Harman et al., 2011).

WBE has also been proposed as a tool for measuring endogenous biomarkers of wellbeing or illness (Choi et al., 2018; Daughton, 2012; Daughton, 2017; Thomas and Reid, 2011). However, the only successful realisation of this concept to date has been the employment of 8-isoprostaglandin F2α (PGF2α) in WBE as a biomarker of oxidative stress. PGF2α load in wastewater correlated with the nicotine metabolite trans-3-hydroxycotinine, indicating an association between oxidative stress and tobacco use (Ryu et al., 2016). WBE otherwise lacks any established endogenous markers of stress.

Multiple criteria exist for a small molecule to be a meaningful WBE biomarker (Daughton, 2018; McCall et al., 2016; Thai et al., 2014a). Among the main considerations are stability and sufficiently high concentrations to allow for effective quantification. A biomarker should be reasonably stable under sewer conditions to be used quantitatively (McCall et al., 2016; O'Brien et al., 2017; Thai et al., 2014b). This can be assessed using laboratory-scale sewer reactors (Gao et al., 2017; Guisasola et al., 2008; Jiang et al., 2009) Degradation of small molecule biomarkers in these systems have proven to be generally representative of real sewer conditions (Li et al., 2018). Additionally, high in-sewer concentrations can allow for cheaper, simpler and faster quantification; samples may not have to be concentrated. Established techniques such as solid-phase extraction (Kasprzyk-Hordern et al., 2008) or more specific immunoaffinity methods (Ryu et al., 2015) are time- and resource-intensive and can introduce additional analytical uncertainties.

The aim of this study was to establish an analytical method for measuring both endogenous (MIAA, histamine metabolite) and exogenous (pharmaceuticals used to treat histamine mediated inflammation) markers that allow an assessment of histamine mediated inflammation and response to histamine mediated inflammation in a catchment population. Furthermore we aimed to identify any differences between catchments and any relationships between these biomarkers. Lastly we evaluated the stability of the biomarkers under different sewer conditions and evaluated whether the stability may have affected the data. To our knowledge, this is the first WBE study to find an interaction between a biomarker of disease and biomarkers(s) of treatment, and the first study of an endogenous biomarker not directly related to oxidative stress or hormones.

2. Materials and methods

2.1. Materials and standards

Analytical grade methanol (MeOH) was purchased from Merck Pty

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