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Mechanism of tyramine adsorption on Ca-montmorillonite

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HIGHLIGHTS

GRAPHICAL ABSTRACT

- Adsorption of tyramine on montmorillonite SAz-2 was dominated by cation exchange.
- Tyramine adsorption capacity was high but limited to 0.57 CEC of the clay.
- Intercalation of hydrated tyramine into the clay interlayer did not cause swell-ing.
- Maximum tyramine loading could be limited by charge-density and steric effects.
- Montmorillonite can be an effective host for tyramine in aquatic environments.



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ABSTRACT

Tyramine (TY) adsorption on a Ca-montmorillonite (SAz-2) was investigated with batch experiments and complementary analyses utilizing ultra-high performance liquid chromatography, ion chromatography, X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, and thermogravimetry (TG). The adsorption reached equilibrium in 8 h, complying with the pseudo-second-order rate equation, and came to an adsorption capacity of 682 mmol kg⁻¹ at pH 6–8.1, utilizing the Langmuir isotherm model. The adsorption of TY and desorption of exchangeable cations exhibited a linear relationship with a slope of 0.9, implying that the adsorption was largely influenced by a cation exchange mechanism. The effective adsorption was further verified by the characteristic TV bands in the FTIR spectra and the signals of mass loss due to TV decomposition in the TG measurements of the clay after adsorption experiments. Intercalation of hydrated TY into the clay interlayer was confirmed by XRD and TG analyses of the heated samples loaded with TY. The adsorption reached only 0.57 cation exchange capacity of the clay which was probably limited by the low charge density of TY as compared to the negative charge density of the clay surface and by the steric effects arising from the hydration of TY that increased its molecular size. Adsorption of TY on montmorillonite can make TY more resistant to thermal decomposition and possibly better preserved in aquatic and soil environments.

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1. Introduction

Tyramine (TY) is one of the common biological amines found in fermented protein-rich foods and remains of aquatic animals after decomposition due to microbial enzymolysis (Liu et al., 2012). The presence of TY in foods can cause allergic reactions such as dyspnea, rash, vomiting, and trigger high blood pressure and other illnesses (Beneduce et al., 2010; Stadnik and Dolatowski, 2010). The risk assessment report of biological amines published by the European Food Safety Authority (EFSA) further clarified that TY is the greatest potential risk factor concerning the safety of proteinaceous food (EFSA Panel on

* Corresponding authors. E-mail addresses: atwtj@mail.ncku.edu.tw, (W.-T. Jiang), li@uwp.edu (Z. Li). Biological Hazards (BIOHAZ), 2011). Thus, biogenic amine in fermented food has become a key focus of attention (Ngapo and Vachon, 2017; Ke et al., 2018). Similar to other amines, TY can also be generated in the brewing processes of a variety of alcoholic beverages, and thus its allowable concentration level is recommended in many countries (Soufleros et al., 1998; Capozzi et al., 2012). Histamine and TY have been regarded as two of the most toxic biogenic amines and the latest research proved that TY is more cytotoxic than histamine, causing necrosis of human intestinal cells (Linares et al., 2016).

Biogenic amines can deteriorate organoleptic properties of surface waters by emanating unpleasant taste and odor and their introduction associated with a significant deficit of dissolved oxygen in surface waters may exacerbate inactivity or death of aquatic animals in reservoirs (Osadchyy et al., 2016). Notably high amine concentrations can occur in summer during detrimental algae flourishing and TY is one of the biogenic amines that were detected in the cells of *Scenedesmus acutus* (Rolle et al., 1977). TY has a low decomposition rate in the environment, and its removal from water is not easy even with high temperature treatments (Cardozo et al., 2013). Hence, TY can be accumulated in aquatic environments through long-term contamination processes or a swift release due to a sudden increase of mass animal remains or discarded foods, which poses a threat to human health and environmental economy (Kim et al., 2004).

Since TY can impose negative impacts on human health and water quality, there have been efforts on investigating removal of amines including TY from aqueous solution or wines with effective adsorbents such as carbon nanotube (Sidorenko et al., 2009), protein-modified silica (Vlasova et al., 2011), and sodium-modified zirconium phosphate (Amghouz et al., 2014). Clay minerals, particularly montmorillonite, typically have large surface areas and high cation exchange capacities and are common substances in aquatic environments and thus adsorption of biogenic amines onto clay minerals can potentially have great influences on the concentrations and behaviors of biogenic amines in surface waters. A systematic characterization of the adsorption of TY on montmorillonite not only extend the range of effective adsorbents for the removal of biogenic amines but also potentially further our understanding of the interactions between biogenic amines and clay minerals in aquatic environments. However, such studies have been scarce, mainly in the context of making TY-modified organoclays for nanocomposite production (Rehab et al., 2007; Chang and Hou, 2008) and herbicide adsorption (Celis et al., 2007; Trigo et al., 2010), without systematic adsorption analyses. In order to illustrate molecular interactions upon adsorption, high levels of intercalation of TY and histamine into STx-1 Ca-montmorillonite were achieved utilizing initial concentrations equal to 10 times of the cation exchange capacity of the mineral at pH ~4.5 and 20 °C (Malferrari et al., 2017).

This study carried out detailed adsorption experiments and determined the adsorption capacity and mechanism of TY onto a calcium montmorillonite in aqueous solution under a number of physiochemical conditions. The result highlights high effectiveness of montmorillonite for adsorptive removal of TY from aqueous solution and for dwelling and accumulation of TY in productive aquatic environments through a cation exchange mechanism.

2. Materials and methods

2.1. Materials

Tyramine hydrochloride ($C_8H_{11}NO \cdot HCl$) with a purity greater than 99% (CAS No. 60-19-5) was purchased from Acros Organics Co., Ltd., Taiwan. The compound has a total molecular weight of 173.65 g/mol and aqueous solubility of 50 g L⁻¹. The molecular structure of TY consists of a flexible ethylamino group as a side chain of its phenolic aromatic ring and a hydroxyl group in the *para* position (Fig. 1a), having a molecular size of $6.3 \times 4.3 \times 4.2$ Å in its gauche conformation and a greater length in the anti-conformation (Amghouz et al., 2014). It has



Fig. 1. Molecular structure (a) and pH-dependent speciation (b) of TY in which α_0 , α_1 , and α_2 are the fractions of TY species in protonated, zwitterionic, and anionic forms, respectively.

a melting point of 164.5 °C (Martha et al., 1983) and a molecular weight of 137.18 g/ mol and was reported to have a pKa value of 9.5 (Vlasova et al., 2011) or $pK_{a1} = 9.3$ and $pK_{a2} = 10.9$ (Perrin, 1965) when dissolved in water (Fig. 1b).

The calcium montmorillonite (SAz-2) used in this study was obtained from the Source Clays Repository of the Clay Minerals Society. The sample received was in a chunky form and was crushed to powder screened with a standard test sieve of 200 mesh (74 µm) without further purification. SAz-2 which is a successor of source clay SAz-1 montmorillonite contains 95–100% montmorillonite and 1–2% quartz with a chemical composition of 60.4% SiO₂, 17.6% Al₂O₃, 0.24% TiO₂, 1.42% Fe₂O₃, 0.10% FeO, 0.08% MnO, 6.46% MgO, 2.82% CaO, 0.06% Na₂O, 0.19% K₂O, 0.02% P₂O₅, and 0.29% F (weight basis) (Chipera and Bish, 2001; Mermut and Cano, 2001). Its cation exchange capacity (CEC) and specific surface area (SSA) were 1200 meq kg⁻¹ (http://www. clays.org/sourceclays_data.html) and 78 m² g⁻¹ analyzed by the Brunauer-Emmett-Teller method using nitrogen gas in this study.

2.2. Batch TY adsorption experiments

To each 50 mL centrifuge tube, 0.1 g of SAz-2 and 10 mL of TY solution were added. All tubes were wrapped with aluminum foils to prevent light-induced decomposition of the compound. For all experiments, except the isotherm study, the initial TY concentration was 2.91 mmol L^{-1} . The final solution pHs were lower than the pK_{a1} value of TY, except the pH effect study. For the kinetic study, the mixing time was 0.25, 0.5, 1.0, 2.0, 4.0, 8.0, 16.0 and 24.0 h. For pH-dependent adsorption experiment, the equilibrium solution pH was between 2 and 11 adjusted by adding 2 mol L^{-1} NaOH or 2 mol L^{-1} HCl drop-

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