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Adsorption isotherms and kinetics for the removal of triclosan and methyl triclosan from wastewater using inactivated dried sludge

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ABSTRACT

Biosorption equilibrium and kinetics of TCS and MTCS sorption to inactivated dried sludge were studied in a batch system with respect to pH, temperature and sludge concentration. Linear and Freundlich isotherms were able to describe the adsorption system well. Sorption capacity increased from 4.80 to 7.36 μg/g and 6.28 to 7.76 μg/g for TCS and MTCS, respectively with an increase in temperature from 15 to 35 °C. As pH decreased from 11.0 to 5.0, the sorption capacity of TCS and MTCS enhanced from 4.36 to 7.92 μg/g and 5.52 to 7.52 μg/g, respectively. The distribution coefficient for MTCS was higher than that for TCS due to hydrophobicity. The calculated K_f and 1/n implied that the adsorption process was physiosorption and exothermic in nature involving weak forces such as Van der Waal's interactions. In the kinetic study, adsorption of TCS and MTCS to dried sludge predominantly followed a pseudo-second order kinetic over the range of applied initial concentrations based on regression coefficients and the relative error for the calculated equilibrium sorption capacity. IR analysis of sludge showed the presence of various polar groups that could highly affect the sorption of organic pollutants.

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1. Introduction

Wastewater treatment plant is basically designed to remove solids, organic matter, pathogens and sometimes nutrients from wastewater before they are released to waterways. Other pollutants present in the wastewater are also affected by the treatment based on their solubility, volatility and degradability. Triclosan (Table 1S, SI) as a broadband antimicrobial agent in healthcare products has been ubiquitously found in wastewater due to its worldwide production and ultimate transfer from household/industrial sewage stream [1]. Researchers estimate that global production of TCS is over 1500 tons per year by which Europe participates in 23.3% of the total production [2]. Widespread production and use of TCS has led to its pervasive occurrence in the environment. This chemical has been frequently found in surface water [3], wastewater [4], sediment [5], human fluids [6,7], fish [8] and etc. Endocrine disruption effect of TCS has been demonstrated so far [9–12]. Moreover, adverse effect of TCS on the aquatic livings and the microbial community has been reported vastly [9,13–17]. Once in the wastewater treatment plant, TCS undergoes mainly adsorption to the sludge and biodegradation due to low solubility and fair biodegradability [18,19] leaving a fraction to be discharged to the environment. The result of microbial degradation of TCS in the biological process is the formation of methyl triclosan (MTCS, Table 1S, SI) which is known as a more persistent compound than parent molecule. MTCS tends to bioaccumulate in the sludge and the living organisms [20]. This compound has been widely detected along with TCS in the environmental samples such as estuarine water and solid [21], soil [22], fish [4] and etc.

Sludge is hugely generated as a by-product of wastewater treatment process and has shown to have high capability for adsorption of organic pollutants from wastewater [23–25]. Constituents in wastewater such as trace metals [23] and organic pollutants [24] are prone to adsorption to the sludge according to the nature of the sludge composition and the interaction involved. Therefore, attempt to improve the sorption capacity of sludge seems promising and advantageous over other type of adsorbents. Most of the newly introduced adsorbents are neither economical nor applicable to real conditions, while sludge is abundant, cheap and easy to use.

Sorption of TCS and MTCS to the sludge is a natural phenomenon, which may allow easier removal of the two compounds from wastewater. A majority of the researches are focused on innovative treatment technologies for TCS removal from wastewater and less attention has been paid to the capacity of dried inactivated sludge for TCS and MTCS removal and description of sorption

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Table 1
FT-IR spectrum interpretation for dried inactivated biomass.

Wavenumber (1/cm)	Mode type	Proposed functional group
3200–3500	Stretching O—H	OH in polymeric compounds
2928	C—H Asymmetric stretching Vibration of CH_2	CH in aliphatic compounds
2850	C—H Symmetric stretching Vibration of CH_2	CH in aliphatic compounds
1630–1670	Stretching vibration of COO	Peptidic bond of proteins
1560	Stretching vibration of C—N and deformation vibration of N—H (amide II)	Peptidic bond of proteins
1460	Deformation vibration of CH_2	
1400	Stretching vibration of C=O	Carboxylates
1230	Deformation vibration of C=O Carboxylic acids Stretching vibration OH	Carboxylates Phenols
1040	C—O Stretching	Ether-like compounds
<1000		Phosphate and sulfur compounds

chemistry and kinetics of the compounds uptake process and still an informational gap about the involved mechanisms exists. Furthermore, although MTCS as a biodegradation product of TCS has been detected along with TCS in the wastewater treatment, no data is so far available about the sorption process in connection to TCS. In this regard, adsorption theory is a useful concept to describe the behavior of TCS and MTCS, and the interaction mechanism between the adsorbate and the biomass. Moreover, it can give useful information about how efficient the sludge may be to remove TCS and MTCS from wastewater.

In this work, attempt was done to investigate the sorption behavior of TCS and MTCS to the dried inactivated sludge. Effect of pH, temperature and amount of adsorbent were studied in a batch scale system. The sorption data were fitted to isotherm models and the most appropriate model was chosen. Moreover, kinetics study was carried out and parameters representing the sorption system and the adsorbate-adsorbent interactions were obtained and discussed.

2. Experimental

2.1. Equilibrium study

Three well-widely used isotherms, Linear, Freundlich and Langmuir were applied to fit the adsorption data. In order to facilitate isotherms analysis, the linearized equations were considered (Eqs. (1)–(3)).

Linear isotherm equation:

$$qe = KdCe \quad (1)$$

Linearized Freundlich equation:

$$\log qe = 1/n \log Ce + \log Kf \quad (2)$$

Linearized Langmuir equation:

$$Ce/qe = 1/KL + aL/KLCe \quad (3)$$

where q_e is the mass of adsorbate per unit mass of adsorbent (μg/g), C_e is the equilibrium concentration of adsorbate in solution (μg/l), K_d is the dissociation coefficient (l/g). n is the empirical Freundlich parameter related to the intensity of sorption. a_L is the Langmuir constant related to the heat of adsorption (l/g). K_f and K_L are the constants of Freundlich (mg/g)(l/mg)$^{1/n}$ and Langmuir (l/mg) models, respectively.

2.2. Kinetic study

The pseudo first-order and pseudo second-order model were employed for the kinetic study according to Eqs. (4) and (5).

Pseudo first-order:

$$\log(qt - qe) = \log qe - k1t/2.303 \quad (4)$$

Pseudo second-order:

$$t/qt = 1/k2qe2 + 1/qet \quad (5)$$

where q_t (μg/g) is the mass of adsorbate per unit mass of adsorbent at time t. k_1 (1/min) and k_2 (g/μg.min) are the rate constants of pseudo first- and pseudo second-order adsorption.

2.3. Reagents and chemicals

TCS was purchased from Wellington Laboratories (Ontario, Canada). MTCS was provided by Dr. Ehrenstorfer GmbH. Dichloromethane (DCM) and ethyl acetate (EA), were from ACS Chemical Inc. Nonane and *n*-hexane were from Anaquachemicals Supply. Methanol (MeOH) was obtained from TEDIA Company Inc. Anhydrous sodium sulfate, sodium hydroxide and formic acid were of analytical grade.

2.4. Preparation of adsorbent

Batch experiments were performed to study the adsorption of TCS and MTC on dried inactivated biomass. For this purpose, active sludge samples from secondary sedimentation of Sha Tin wastewater treatment plant were collected in amber glass jars. Plant specification is presented in Table 2S (SI). The samples were stored in an ice-filling cooler to be delivered to the analytical laboratory. 1% MeOH (v/v) was immediately added to the samples to restrain the microorganism's activity and biodegradation [26]. Samples were centrifuged at 6000 rpm for 8 min. Since the sludge sample initially contained TCS and MTCS, the samples were sonicated with sequential 3 aliquots of 50:50 MeOH:H_2O solution to ensure complete extraction of target compounds. Then, the samples were further lyophilized in the freeze-dryer. A portion of biomass was analyzed separately for the presence of any target compound residue and background interference. The concentration of TCS and MTCS in this sample had to be below the MDLs, otherwise correction of concentration was applied.

2.5. Development of an analytical method for determination of TCS and MTCS

An analytical method was developed to determine the adsorbed amount of analyte. 100 ml pre-determined wastewater sample was spiked with 2.0 μg TCS and 1.0 μg MTCS and shaken for 2 h in a water-bath shaker. The solution was then acidified with 100 μl formic acid. SPE cartridge, Isolute Env+ was applied for simultaneous extraction and cleanup. For this purpose, the column was mounted on a 12-port vacuum manifold (Alltech, Deerfield U.S.A) and conditioned with sequential 6 ml aliquots of EA, MeOH and 1% acidified DI water, respectively with a flow rate of 2 ml/min. Then, the sample solution was loaded onto the cartridge with the same flow rate. Sample container was rinsed twice with 5 ml MeOH/H_2O 5% (v/v) and added to the SPE column as washing step. The column

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