Adsorption of dimethyl trisulfide from aqueous solution on a low-cost adsorbent: thermally activated pinecone*

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Received Apr. 9, 2014; accepted in principle May 28, 2014; accepted for publication Jun. 23, 2014 © Chinese Society for Oceanology and Limnology, Science Press, and Springer-Verlag Berlin Heidelberg 2015

Abstract Thermally activated pinecone (TAP) was used for the adsorption of dimethyl trisulfide (DMTS) from aqueous solutions, which was proved to be the main odorous in algae-caused black bloom. The effects of adsorbent dosage, adsorbate concentration and contact time on DMTS biosorption were studied. The TAP produced at 600° C exhibited a relatively high surface area (519.69 m²/g) and excellent adsorption capacity. The results show that the adsorption of DMTS was initially fast and that the equilibrium time was 6 h. Higher initial DMTS concentrations led to lower removal percentages but higher adsorption capacity. The removal percentage of DMTS increased and the adsorption capacity of TAP decreased with an increase in adsorbent dosage. The adsorption process conforms well to a pseudo-second-order kinetics model. The adsorption of DMTS is more appropriately described by the Freundlich isotherm (R^2 =0.996 1) than by the Langmuir isotherm (R^2 =0.916 9). The results demonstrate that TAP could be an attractive low-cost adsorbent for removing DMTS from water.

Keyword: dimethyl trisulfide; low-cost; adsorption isotherms; adsorbent

1 INTRODUCTION

Sulfides are most commonly identified notable taste and odor (T&O) chemicals in algae-induced black bloom. For example, dimethyl trisulfide (DMTS) was detected and proven to be the main odorous compound in Wuxi's water crisis in the summer of 2007 (Lloyd et al., 1998; Lu et al., 2013). The chemicals responsible for T&O were cause by the anaerobic decomposition of algae (Yang et al., 2008; Li et al., 2012). DMTS cause taste and odor at concentrations as low as 10 ng/L. Therefore, the treatment methods for T&O compounds must be highly effective. DMTS is negligibly removed by conventional treatment processes due to their low molecular weights. Various methods, including coagulation / flocculation, adsorption, biosorption, advanced oxidation and ozonation, have been employed for the treatment of T&O from water (Cook et al., 2001; Wakita et al., 2001; Park et al., 2007; Muthuraman et al., 2011; Zhang et al., 2011). Among various treatment methods, adsorption is more effective than other techniques because of its cost-effective, simplicity of design, and ease of operation. Activated carbon is usually a preferred adsorbent because it has high adsorption capacity for a large number of organic/inorganic metal ions and a high BET surface area (Nowack et al., 2003; Drikas et al., 2009). However, the high cost of activated carbon technologies prevents their use in applications. Therefore, developing an easily handled and cost-effective adsorbent is necessary.

Low-cost agricultural wastes are the most promising materials being studied for this purpose

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^{*} Supported by the Taihu Special Project of Water Pollution Control, Jiangsu Province (No. TH2013214), the National Water Pollution Control and Management Technology Major Project (No. 2012ZX07103-005), the Industry-Academia Cooperation Innovation Fund Project of Jiangsu Province (No. BY2011165), and the Open Foundation of State Key Laboratory of Lake Science and Environment, CAS (No.2014SKL005)

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because they are renewable, locally available and cost-effective than other materials (Nunes et al., 2009). The production of biochar from agricultural wastes by thermal-chemical conversion has gained extensive much attention over the last decade. A large number of studies on the use of agricultural waste as an adsorbent to remove heavy metals and dyes have been found in the literature, examples include adsorption of phenol by apricot stone shells (Daifullah and Girgis, 1998); adsorption of methylene blue with coffee press cake (Nunes et al., 2009) or rice husk ash (Chowdhury et al., 2009); removal of pesticides by several low-cost biomass sorbents (Zolgharnein et al., 2011); and removal of heavy metals, such as cadmium (II) or copper, by chemically activated pinecone and other low-cost agricultural biomass (Reddad et al., 2002; Amarasinghe and Williams, 2007; Argun et al., 2008; Arief et al., 2008; Wang et al., 2010; Ofomaja and Naidoo, 2011; Dawood and Sen, 2012). There are a limited number of studies related to T&O adsorption onto biochar (Ng et al., 2002).

The aim of this study was to investigate the possibility of using thermally activated pinecone (TAP) as a low-cost and environmentally friendly adsorbent for DMTS removal from aqueous solution. The adsorption characteristics of DMTS onto TAP were investigated under varying conditions, such as different contact times, adsorbent dosages, and initial DMTS concentrations. To understand the adsorption both kinetic mechanism, and equilibrium measurements were performed and complemented with scanning electron microscopy (SEM) analyses and Fourier transform infrared spectroscopy (FTIR).

2 MATERIAL AND METHOD

2.1 Adsorbent preparation and regeneration

Pinecones were washed three times with distilled water to remove ash and impurities and then oven-dried overnight at 100°C. The dry pinecones were then ground using a crusher and passed through a 200-mesh sieve. The thermal activation procedure of TAP was established based on the method of the production of crop residue-derived chars by thermal activation (Chun et al., 2004). The pinecones were subjected to thermal activation at 200, 400, 500, and 600°C for 6 h under oxygen-limited conditions. The resulting residues were then treated in 500 mL of 1 mol/L HCl for 12 h and centrifuged to remove the supernatants. Finally, the samples were washed with distilled water three times and dried at 100°C for 24 h.

SEM observations of the TAP were performed with a Zeiss EVO 18 SEM. BET surface area was measured by the nitrogen adsorption method using a Tristar3020 surface area and porosity analyzer (Micromeritics Instrument Corp., USA). Changes in the surface structures of pinecones, before and after thermal activation, were examined using FTIR spectroscopy with a Nicolet iS10 FT-IR spectrometer (Thermo Scientific). The spectra of adsorbents were measured within the range of 500–4 000/cm wave numbers.

The thermal regeneration was done by heating the exhausted TAP at 105°C for 12 h.

2.2 Preparation of DMTS solution

DMTS stock solution was prepared (2 000 mg/L) by dissolving the odorous compound in CH₃OH (HPLC grade, \geq 99.9% purity). The stock solution was stored at 4°C to avoid volatilization. All the working solutions were prepared with deionized water. The properties of the DMTS are given as follows: molecular formula: C₂H₆S₃, average mass: 126.264, Log Kow=1.87, boiling point: 58°C, density: 1.202 g/mL at 25°C.

2.3 SPME and GC conditions

The concentration of DMTS in the solution was analyzed by headspace solid-phase microextraction (HS-SPME) and an Agilent 7890A gas chromatograph coupled with flame photometric detector (GC-FPD) sulfur mode. A 75-µm Carboxen/polydimethylsiloxane (CAR/PDMS)-coated SPME fiber (Supelco, Bellfonte, PA) was used to capture DMTS. The sample (20 mL) was added to a 40-mL, screwcapped HS-SPME vial with a PTFE-faced silicone septum. The sample was stirred with a magnetic hot stirrer (60°C) at 500 r/min using PTFE-coated magnetic stirring bars. The outer needle of the fiber was passed through the septum and the fiber extended into the headspace above the sample for 30 min. After extraction, the fiber was placed into the injector of the GC and desorbed for 3 min at 250°C. GC conditions were as follows. The column was an Agilent J&W GS-GasPro GC Column (60 m×0.32 mm I.D). The temperature of the injector and detector was set to 250°C. The carrier gas was He (99.999%) at a constant flow rate (3 mL/min), and it was supplied with 50 mL/ min of H₂, 65 mL/min of synthetic air and 30 mL/min of N₂ as auxiliary gas mixture. The temperature program consisted of an initial run at 50°C for 5 min followed by a gradient of 25°C/min up to 250°C and then temperature hold for 10 min at 250°C (Lu et al., 2012).

2.4 Adsorption studies

All adsorption experiments were conducted using similar batches of starting materials with known amounts of adsorbent and 40 mL of aqueous DMTS solution in glass flasks. The glass flask was sealed with a PTFE/silicon septum-fitted screw cap. The mixture was shaken at a constant temperature (25°C) on a temperature-controlled shaker at 150 r/min.

A series of adsorption experiments was performed. In the first set of experiments, the adsorption of DMTS by natural pinecones (100°C), TAP (activated at 200, 400, 500, and 600°C) and powder activated carbon (Sinopharm Chemical Reagent Beijing Co., Ltd.) (PAC) was investigated for comparison. DMTS solution (40 mL, 4 µg/L) and 10 mg adsorbent were mixed into glass flasks and shaken for 12 h. The second set of experiments was designed to study the effect of adsorbent dosage on the DMTS removal. Different quantities of adsorbent, for example 1, 2, 4, 8, 10, and 20 mg, were added into the flask containing DMTS solution (40 mL, 4 µg/L) and stirred for 12 h. The third was designed to investigate the effect of adsorbent contact and equilibrium time. DMTS solution (40 mL, 4 µg/L) and 1 mg adsorbent were mixed and stirred for periods of time ranging from 0.5 to 6 h. The adsorption isotherm and the effects of initial DMTS concentration were also studied in the present experiment. DMTS solutions of 2, 4, 8, 22, and 33 µg/L were prepared and then treated with 1 mg adsorbent for 12 h. To make the adsorption process more economical, it is necessary to regenerate the TAP. After the attainment of equilibrium, the adsorbent was washed with 1 mol/L HCl (guaranteed reagent) for 6 h and dried at 105°C overnight. The adsorption of DMTS ($40 \,\mathrm{mL}, 4 \,\mathrm{\mu g/L}$) by regenerated TAP ($10 \,\mathrm{mg}$) was investigated.

All samples were filtered through 0.45 μm filter membrane before analysis. Blank samples not containing adsorbent were also prepared and analyzed using the same procedures.

2.5 Removal efficiency and the adsorption capacity

The removal efficiency of DMTS (% adsorption) was calculated as:

Removal efficiency (%) =
$$\frac{C_0 - C_t}{C_0} \times 100$$
, (1)

and the amount of DMTS adsorbed onto the adsorbent at time t is q_t (µg/g), which was calculated as the following:

$$q_t = \frac{(C_0 - C_t)}{m} V \,, \tag{2}$$

where C_0 and C_t are the concentration of DMTS (μ g/L) at initial and any time t; V (L), and m (g) are the volume of solution and the mass of adsorbent, respectively.

2.6 Adsorption kinetics and isotherm

The pseudo-first-order (Eq.3) (Reddad et al., 2002) and pseudo-second-order models (Eq.4) (Kumar and Sivanesan, 2006) were used to investigate the adsorption kinetics of DMTS onto TAP:

$$q = q_{\rm e}(1 - {\rm e}^{-k_1 t})$$
, (3)

$$q = \frac{k_2 q_{\rm e}^2 t}{1 + k_2 q_{\rm e} t},\tag{4}$$

where q_e (µg/g) and q_t (µg/g) are the amount of adsorbate adsorbed per gram of adsorbent at equilibrium and at any time t (min), respectively. k_1 (/min) and k_2 (g/(µg·min)) are the rate constants of the pseudo-first-order and pseudo-second-order adsorption, respectively.

The adsorption equilibrium data of DMTS on TAP were tested with Langmuir (Eq.5) and Freundlich isotherm models (Eq.6):

$$q_{\rm e} = \frac{q_{\rm max}bC_{\rm e}}{1 + bC_{\rm e}},\tag{5}$$

$$q_{\rm e} = k_{\rm f} \times C_{\rm e}^{\frac{1}{n}} , \qquad (6)$$

where $C_{\rm e}$ is the equilibrium concentration of the solute $(\mu {\rm g/L})$, $q_{\rm e}$ is the amount of solute adsorbed per gram of TAP $(\mu {\rm g/g})$, b $({\rm L/\mu g})$ is the Langmuir constant related to the adsorption energy, $q_{\rm max}$ $(\mu {\rm g/g})$ is the maximum adsorption capacity, $k_{\rm f}$ $(\mu {\rm g/g})$ $({\rm L/\mu g})^{1/n}$ and n are the Freundlich constants that indicate the adsorption capacity and intensity, respectively.

3 RESULT AND DISCUSSION

3.1 Influence of activated temperature

Removal efficiency of DMTS by TAP and untreated pine conespinecones were compared (Fig.1). Thermal activation provided an improvement in the adsorbing ability, with a significant increase in the removal efficiency when activated at 400°C (75.42±6.21)%. The TAP activated under 600°C had the highest adsorbing capacity (98.93±2.99)% when compared with other activation temperatures and activated

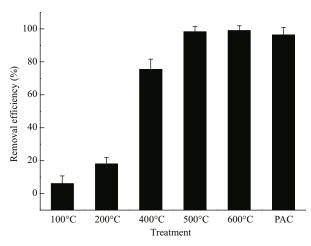


Fig.1 The removal efficiency of DMTS by adsorption onto different adsorbents

Thermally activated pinecone at different temperatures and activated carbon.

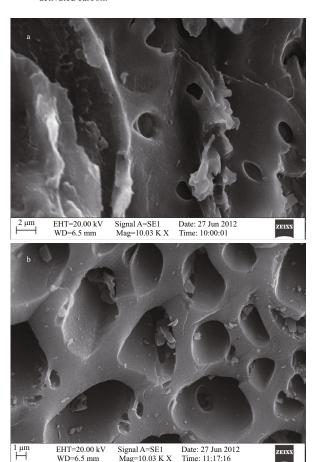


Fig.2 SEM image of (a) pinecone and (b) thermally activated pinecone

carbon (96.42±4.32)%. The results indicate that TAP activated at 600°C is a more effective adsorbent for the removal of DMTS than conventional activated carbon. So in this study, TAP (thermal activation at 600°C for 6 h) was further selected for the remaining

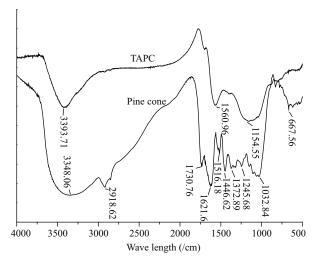


Fig.3 The surface characteristics of TAPC measured by FT-IR spectroscopy

tests. The SEM enables a direct observation of the surface microstructures of samples. SEMs of pinecone before and after thermal activation are shown in Fig.2a and 2b, respectively. The availability of pores and interior surfaces can be seen clearly in the SEM image.

The activation temperature has a significant effect on surface area and on external and internal pore formation or collapse (Nunes et al., 2009). For example, the increase in BET surface area ranged from 2.96 m²/g to 519.69 m²/g. This increase in surface area for the TAP could be attributed to thermal activation and thus the availability of pores (Fig.2b). A higher BET surface area is thought to be one of the main reasons for higher adsorption capacity of TAP (600°C) for DMTS.

The FTIR spectra of nature and TAP thermally activated pinecone within the range of 500-4 000/cm wave numbers were measured (Fig.3). For example, the absorption peak of approximately 3 348.06/cm indicates the existence of free and intermolecularly bonded -OH groups in natural pinecone (Dawood and Sen, 2012). The peaks observed at 2 918.62/cm can be assigned to the stretching vibration of the C-H group (Ofomaja and Naidoo, 2011). The peaks of approximately 1 621.6/cm correspond to the C=C stretching that confirms the presence of the lignin aromatic groups. The C-O band at 1 032.8/cm is also attributed to the presence of lignin. The peak at 667.7/cm can be attributed to bending vibrations of aromatic compounds (Garg et al., 2007). In TAP, an absorption band is observed at 3 393/cm can be assigned to the bonded -OH groups. The other prominent peaks are due to -OCH₃ group and C-O-C.

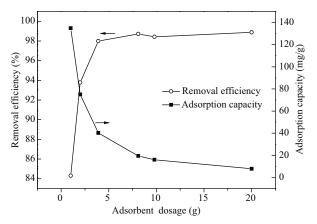


Fig.4 The effect of adsorbent dose on the removal and adsorption of DMTS by TAPC

The results show that following heat treatment, the intensity of -OH groups absorption peak in the infrared absorption spectrogram decreased significantly.

3.2 Influence of adsorbent dosage

The impact of adsorbent dosage on DMTS adsorption in TAP was investigated with an initial concentration of 4 µg/L. The removal efficiency of DMTS increased when the adsorbent dosage were increased. After 4 h, the removal of DMTS varied from 74% to 98% with increasing adsorbent dosage from 1 to 20 mg, respectively. This removal efficiency enhancement could be attributed to increased adsorbent surface area and availability of more adsorption sites resulting from the increase in TAP dosage (Dawood and Sen, 2012). However, the results also demonstrated that the decrease in adsorption capacity (uptake of per gram of adsorbent) is mainly due to unsaturation of adsorption sites on TAP through the adsorption process (Nunes et al., 2009; Wang et al., 2010; Dawood and Sen, 2012). Another possible reason may be due to the particle aggregation, resulting from high TAP dosage. The total surface area of the adsorbent would be decreased under such particle aggregation (Garg et al., 2007; Wang et al., 2010). Based on these results (Fig.4), the remaining experiments were conducted at an adsorbent dosage of 1 mg.

3.3 Effect of contact time

The effect of contact time on the adsorption of DMTS was investigated with the time exposure ranging from 0.5 to 8 h, and the results are presented in Fig.5.

The results in Fig.5 demonstrated that the removal of DMTS by adsorption on TAP was extremely fast initially; about 63% of DMTS was removed within 60 min. Thereafter, the removal efficiency increased

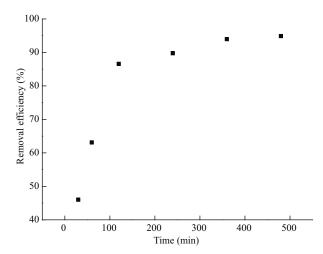


Fig.5 The effect of the contact time on the adsorption of DMTS by TAPC

slowly. The equilibrium is attained within 6 h. Similar behavior of the effect of contact time on T&O adsorption capacity has been reported in the literature for other types of adsorbents (Garg et al., 2007; Cook et al., 2001). The initial period with a rapid removal rate is apparently attributed to the increase of abundance of available free adsorption sites on TAP. After this initial period, molecules of DMTS must diffuse deeper into the micropores of the adsorbent, which subjects DMTS to a larger resistance between the aqueous and solid phases (Wang et al., 2011). Therefore, adsorption of DMTS by TAP slows down with time.

3.4 Adsorption kinetics and isotherms

The prediction of adsorption kinetics is necessary because it evaluates parameters that are critical in the design of industrial adsorption columns (e.g., by predicting the $q_{\rm e}$ values and estimating adsorption rates, etc.). In the present study, the pseudo-first-order (Eq.3) and pseudo-second-order model (Eq.4) were used to analyze the experimental data.

The results of the kinetic parameters of adsorption, including the k_1 (pseudo-first-order rate constant), k_2 (pseudo-second-order rate constant), $q_{\rm e,cal}$ (µg/g) (calculated equilibrium adsorption capacities) and regression coefficients (R^2), are listed in Table 1. The value of linear correlation for the pseudo-second-order kinetic model (0.990 7) was higher than that of the pseudo-first-order kinetic model (0.976). The $q_{\rm e,cal}$ using calculated from the pseudo-second-order model is in agreement with very closer to the experimental $q_{\rm e}$. This result indicated that the adsorption of DMTS onto TAP follows the pseudo-second-order kinetic model very well, which is in agreement with many previous studies (Wang et al., 2011; Dawood and Sen, 2012).

Table 1 Pseudo-first-order and pseudo-second-order parameters for the adsorption of DMTS by TAPC

C ₀ (μg/g)	q _e (μg/g)	First-order kinetics			Second-order kinetics		
		$k_1(/\text{min})$	$q_{ m e,cal}(\mu{ m g}/{ m g})$	R^2	k ₂ (g/(μg·min))	$q_{\rm e,cal}(\mu{ m g/g})$	R^2
4	235.37	0.023 9	230.2	0.976	0.000 14	252.6	0.990 7

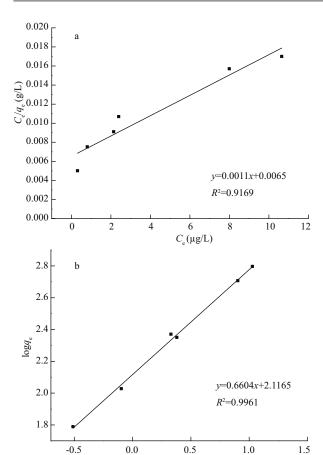


Fig.6 The adsorption parameters from the Langmuir (a) and Freundlich isotherms (b)

 $logC_{a}$

Adsorption isotherms are crucial for understanding the mechanism of how the DMTS interacts with the TAP and gives an idea of the adsorption capacity of the adsorbent. The experimental data were tested with Langmuir and Freundlich isotherms(Fig.6). A detailed analysis of regression coefficients showed that the Freundlich isotherm (R²=0.997 5) had a better fit than the Langmuir isotherm ($R^2=0.9004$) (Table 2). It has been proved that the constant of n gives an indication of the favorability of the adsorbent. The n of Freundlich isotherm models in the present study is larger than 1, indicating that DMTS is preferentially adsorbed onto the adsorption sites of TAP and a physical process (Annadurai et al., 2000). Similar results have been reported for the removal of dimethyl sulfide by bamboo charcoal (Wang et al., 2011).

Table 2 Isotherm model parameters for the adsorption of DMTS by TAPC

Equilibrium model	Parameter	Value	
	$k_{\rm f} \; (\mu {\rm g/g}) \; ({\rm L/\mu g})^{\rm 1/n}$	131.58	
Freundlich isotherm	1/n	0.657	
	R^2	0.997 5	
	$q_m(\mu g/g)$	1 098	
Langmuir isotherm	<i>b</i> (L/μg)	0.117	
	R^2	0.900 4	

Table 3 The influence of adsorption-regeneration on adsorption capacity

Adsorption-regeneration cycle	0	1	2	3
Adsorption capacity decrease %	0	4.52	4.77	8.79

3.6 Regeneration

To make the adsorption process more economical, it is necessary to regenerate the adsorbent. The adsorption-regeneration cycle of DMTS was repeated three times. Table 3 demonstrates that the adsorption capacity drops slightly during the first two cycles. Even after the third adsorption-regeneration cycle, the adsorption capacity of TAP declined by only 8.78%. These results suggest that thermal regeneration is feasible and can be easily carrying out, then reducing the operation cost.

4 CONCLUSION

Given the high removal efficiency and the low cost, adsorption with agricultural waste seems to be an economic method for the treatment of T&O from the contaminants water. In the present study, TAP, a common agricultural waste, were uses as an alternative low cost adsorbent to remove DMTS. The DMTS removal was highly dependent on initial concentration, TAP dosage and contact time. The DMTS adsorption on the adsorbent is better described by the Freundlich adsorption isotherm than the Langmuir isotherm. The kinetic studies showed that the DMTS adsorption process followed a pseudo-second-order kinetics model. Even after three adsorption-regeneration cycles, TAP still have a high adsorption capacity. In summary, the results presented in this study suggest

that TAP would be a promising low-cost adsorbent for the removal of DMTS.

5 ACKNOWLEDGEMENT

We thank the Taihu Laboratory for Lake Ecosystem Research (TLLER), Nanjing Institute of Geography and Limnology, Chinese Academy of Science. We sincerely thank Dr. ZHANG Lei and Dr. LU Xin for their friendly help during our experiments.

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