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Research article

# Arsenite and arsenate removal from wastewater using cationic polymer-modified waste tyre rubber

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ABSTRACT

Waste tyre rubber (WTR) granulate was modified with a cationic polymer, poly(3-acrylamidopropyl) trimethylammonium chloride (p(APTMACl)). The resulting WTR/p(APTMACl) was utilized for the adsorption of arsenite, As(III) and arsenate, As(V) from aqueous medium in both batch and column methods. The level of adsorption increased gradually with increasing monomer concentration and contact time. The adsorption behavior obeyed the Freundlich model, and the rate of adsorption could be predicted by employing the pseudo-second order model. In the column method, As(V) could be adsorbed onto the sorbent more effectively than As(III). Remarkable desorption of As(III) and As(V) (99 and 92%, respectively) from the adsorbent was achieved using 0.10 M HCl as eluent. An approach of evaluation of adsorption capacity uncertainty is proposed.

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## 1. Introduction

Arsenic (As) has been used in various industrial fields including agriculture, electronics, and metallurgy. The toxicity of arsenic to human health ranges from skin lesions to cancer of the brain, liver, kidney, and stomach (Liao et al., 2009). Arsenic has been detected in groundwater in several countries, with concentration levels exceeding the World Health Organization (WHO) standard for drinking water (10 µg/L) (WHO, 2014). Therefore, methods for the remediation of arsenic-contaminated water are critical and necessary.

Numerous methods have been used to remediate arsenic-contaminated water such as phytoremediation, chemical oxidation and photochemical oxidation (Singh et al., 2015). However, these methods are fairly sophisticated processes and can be expensive at a large scale. Adsorption is an alternative method. Many researchers have focused on finding low-cost adsorbents. The materials studied are $TiO_2$ (Jegadeesan et al., 2010), chars (Fan et al., 2004), red mud (Sahu et al., 2010), iron oxide-loaded slag (Zhang and Itoh, 2005), waste tyre rubber (Rowley et al., 1984), activated alumina (Singh and Pant, 2004), reclaimed iron-oxide coated sand (Hsu et al., 2008), natural alum silicate exchangers (Habuda-Stanić et al., 2008), and inorganic-organic hybrid adsorbents (Hao et al., 2009).

The amount of waste tyre rubber (WTR) arising from the automobile industry is increasing every year. As a result, the management and disposal of WTR has become a great concern. Finding new ways to reutilize WTR is a challenge. Uses of WTR for the adsorption of heavy metals from water and wastewater have been reported (Alexandre-Franco et al., 2011; Calisir et al., 2009; Danwanichakul et al., 2008; Gupta et al., 2012). In addition, carbon black used to reinforce the rubber, is similar to activated carbon and offers as a good adsorbent. Therefore, in this study we have probed the effectiveness of using WTR as a base material for the preparation of adsorbents for the removal of arsenic from wastewater streams.

Some of the problems associated with the use of WTR are its sorption capacity and slow adsorption kinetics. Therefore, the aim of this work is to improve the efficiency of WTR for arsenic removal by surface-modification using a polymer. Polymers previously used for arsenic adsorption are poly(3-acrylamidopropyl)trimethylammonium chloride (p(APTMACl)) (Barakat, and Sahiner, 2008), poly(methyl acrylate), poly(vinyl acetate), poly(acrylic acid) (Malana et al., 2011), poly(vinyl alcohol) (Santos et al., 2012), quaternized poly(4-vinylpyridine) (Sahiner et al., 2011), and poly(-acrylonitrile-*co*-acrylamidopropyl-trimethyl ammonium chloride)

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(Dudu et al., 2015). Among these polymers, cationic p(APTMACl) has attracted great interest due to its simple formation in aqueous solution. Thus, in this work, WTR granulate was modified with p(APTMACl), and the arsenic adsorption through employing the adsorbent in both batch and column methods were investigated. A column of modified adsorbent utilized to remove arsenic from an industrial wastewater sample has been obtained.

## 2. Experimental

### 2.1. Materials and reagents

(3-Acrylamidopropyl)trimethylammonium chloride (APTMACl, 75 wt % solution in water), *N*,*N*′-methylenebisacrylamide (MBA), *N*,*N*,*N*′,*N*′-tetramethylethylenediamine (TEMED) and ammonium persulfate (APS) were purchased from Sigma–Aldrich. As(V) and As(III) stock solutions, 1000 mg/L were purchased from Sigma–Aldrich. Arsenic concentrations in wastewater samples were measured using an inductively coupled plasma optical emission spectrometer (ICP-OES, iCAP 6500, Thermo Scientific).

### 2.2. Preparation of waste tyre rubber adsorbent

WTR granulate (average diameter of 295 μm, particle size distribution shown in Fig. S1, Supplementary data) was obtained from Union Commercial Development Co. Ltd. (Samut Prakan, Thailand). WTR was surface-modified with APTMACl using the following procedure. Approximately 0.50 g of WTR was mixed with 5 mL of the desired amount of APTMACl monomer (250–2000 mM), 10 mM of TEMED and 50 mM of MBA. Then, the mixture was sonicated for 15 min followed by purging with $N_2$ for 1 h. About 0.50 mL of 70 mM APS solution was added, and the mixture was stirred continuously at 100 rpm for 1 h to complete the reaction. The solid was then separated by filtration. After that, it was washed several times with deionized water and dried for 24 h in an oven at 50 or 100 °C before further use.

### 2.3. Characterization of adsorbents

The functional groups on the surface of WTR and the resulting modified WTR by p(APTMACl) were characterized using Fourier transform infrared spectroscopy (FT-IR, Nicolet 6700, Thermo Scientific) using the attenuated total reflectance (ATR) mode.

### 2.4. Arsenic adsorption

#### 2.4.1. Batch method

0.10 g WTR/p(APTMACl) adsorbent was added into 10 mL of arsenic solution (10 mg/L As(III) or As(V)) in a test tube and the tube was shaken at room temperature for a set time (30 min–24 h). The pH was then adjusted to a specific value (from 3 to 10) by using either 1% (v/v) HCl, or 1% (w/v) NaOH solution. Afterward, the suspension was filtered through a 0.45 μm filter membrane. Arsenic concentrations in the solution, before and after adsorption, were determined using ICP-OES. All experiments were performed in triplicate. The adsorption efficiency of arsenic (mg As/g adsorbent) was calculated.

The adsorption isotherm was evaluated as follows: approximately 0.10 g of WTR/p(APTMACl) was added into individual vials which contain 10 mL of arsenic solution of different concentrations (5–60 mg/L). The mixture was shaken at 60 rpm at room temperature for 24 h. The solid was separated by filtration, and the residual arsenic concentrations were then determined.

#### 2.4.2. Column method

WTR/p(APTMACl) was packed into cylindrical columns (5 mL plastic syringe barrel, id 12.5 mm), each having different absorbent layer heights (1.0, 1.5, and 2.0 cm). Then, 25 mL of 10 mg/L arsenic solution was passed through the column. The effect of several different flow rates on adsorption was studied. In addition, desorption of arsenic was studied with 0.05 M, 0.1 M, and 0.5 M HCl solutions. The breakthrough curves of the column were also studied using arsenic solution in the range of 10–120 mL. Arsenic concentrations in both eluates and eluents were determined.

### 2.5. Arsenic removal from industrial wastewater samples

Two industrial wastewater samples (W1 and W2) were provided by a petroleum company (Thailand). Twenty-five milliliters of each wastewater sample were consecutively passed through columns of 1.5 cm in bed height. The residual arsenic concentrations in the eluate solution were determined.

## 3. Results and discussion

### 3.1. Characterization of adsorbents

FT-IR spectra of WTR and WTR/p(APTMACl) are shown in Fig. 1. Peaks at 2912 and 2840 cm$^{-1}$, and 1450 and 1368 cm$^{-1}$, are assigned to $CH_3$ and $CH_2$ and the peak at 1541 cm$^{-1}$ is assigned to the $COO^-$ stretching of zinc carboxylates in the rubber structure (Zanchet et al., 2012). The small peaks occurring at 875 and 841 cm$^{-1}$ are attributed to crosslinking $-CH_2$-S- groups (Ekici et al., 2011). A characteristic peak at 3200–3400 cm$^{-1}$ due to the presence of water associated in the polymer matrix was observed in the WTR/p(APTMACl) sample. In addition, the appearance of extra peaks at 1210, 1368, 1531, and 1632 cm$^{-1}$ in Fig. 1(a) corresponds to the presence of amide functionalities from the p(APTMACl) polymer (Kaşgöz et al., 2003).

### 3.2. Arsenic adsorption by batch method

#### 3.2.1. Effect of pH

The amount of As(III) and As(V) adsorbed by WTR/p(APTMACl) was evaluated over a pH range of 3–10 as shown in Fig. 2. Amounts of adsorbed As(III) are lower than that of As(V). The level of As(III) adsorption was similar at all pH values. WTR/p(APTMACl) adsorbent contains the $-N^+(CH_3)_3$ functional group which is highly susceptible to ion exchange for anionic species (Mohan and Pittman, 2007). As(III) exists in solution as mainly $H_3AsO_3$, and marginally as $H_2AsO_3^-$ (Sharma and Sohn, 2009). Therefore, only weak adsorption was expected. For As(V) the highest adsorption

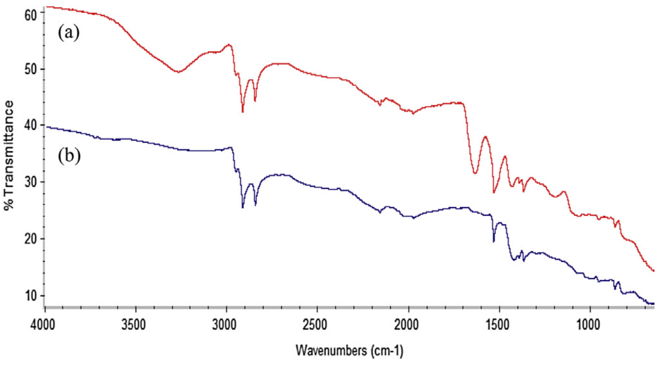

**Fig. 1.** FT-IR spectra of (a) WTR/p(APTMACl) and (b) WTR.

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