



## Research article

## Removal of arsenate and dichromate ions from different aqueous media by amine based p(TAEA-co-GDE) microgels

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## ABSTRACT

In this work, microgels based on tris(2-aminoethyl) amine (TAEA) and glycerol diglycidyl ether (GDE) via simple microemulsion polymerization was prepared as p(TAEA-co-GDE) microgels were used as adsorbent for removal of dichromate (Cr (VI)) and arsenate (As (V)) ions from different aqueous environments. The p(TAEA-co-GDE) microgels were demonstrated very efficient adsorption capacity for Cr (VI), and As (V) that are 164.98 mg/g, and 123.64 mg/g from distilled (DI) water, respectively. The effect of the medium pH on the adsorption capacity of p(TAEA-co-GDE) microgels for Cr (VI) and As (V) ions were investigated. The maximum adsorption capacity was obtained at pH 4.0 for both ions with maximum adsorbed amounts of 160.62, and 98.72 mg/g, respectively. In addition, the microgels were also shown moderate adsorption capacity for Cr (VI) and As (V) from other water sources; tap water with 115.18 mg/g and 82.86 mg/g, sea water with 64.24 mg/g and 46.88 mg/g and creek water with 73.52 mg/g and 59.33 mg/g, respectively. Moreover, the increase in adsorbent dose from 0.025 to 0.125 g enhanced % adsorption of Cr (VI) from 54.13 to 98.03, and As (V) from % 26.72–98.70, respectively. For the adsorption process Langmuir and Freundlich adsorption isotherms were applied and found that Langmuir adsorption isotherm with  $R^2$  value of 0.99 for both the metal ions are suitable. Moreover, the experimental adsorption capacities of Cr (VI) and As (V) were found very close to the theoretical values calculated from Langmuir adsorption isotherm. More importantly, the microgels were made magnetic responsive to recover them easily from adsorption medium for reuse studies by applying external magnetic field with little decrease in adsorption capacity. Additionally, reusability of p(TAEA-co-GDE) microgels was also evaluated for adsorption of Cr (VI) and As (V) from DI water.

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## 1. Introduction

Due to the demands for fast industrial developments in the last few decades, toxic metals contamination of drinking water resources has become a serious problem causing worldwide concern (Atta et al., 2015). Generally these toxic metal ions became a part of the environment by burning fossil fuels and other processes such as mining and smelting (O'Connell et al., 2008). The growth of industrialization, mining, mineralogy, machinery, coal combustions

and other human activities generated numerous environmental problems (Ozay et al., 2009; Srivastava and Majumder, 2008). All these activities by human lead to contamination of water sources. It was reported that about 5 billion people living in underdeveloped regions will lack sufficient clean water by the year 2025 (Rowell, 2006). The issue is even severe in developing countries where metal ion contaminations of water resources cause polluted soil; kill aquatic life and lead to serious health issues in mankind (Guo et al., 2017). There are many ways for metal ion to effect human or living organism e.g., heavy metal ions are accumulated in the different parts of human and other living organisms from sewage water, industrial wastes and ineffective filtration in household

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systems. Due to non-biodegradable nature of metal ions, these severe health problem can occur in different parts of body like liver, kidney, nervous system, bone, teeth, and endocrine gland etc. (Abbas et al., 2016; Ahmed and Ahmaruzzaman, 2016). Heavy metal ions such as Arsenic and Chromate have been of special interest due their very strong toxicity even at very low concentrations (Gao et al., 2009; Sahiner et al., 2015b). Due to their high mobility, these metal ions spread very quickly to large areas and their removal from water is a challenging task for researchers.

Arsenic is abundantly present in atmosphere, natural water resources, rocks and different organism. It enters into aquatic environment from different sources like medicinal activities, mining, erosion, agriculture, industrial pollutants, chemical processing, metals preparation, fertilizers, pesticides, ceramics and glasses (Bayrakci and Yigiter, 2013; Castaldi et al., 2014; Shao et al., 2008). Arsenic is of great concern worldwide due to its high toxicity and carcinogenicity. It is present in natural form in earth crust with no detectable taste and odor. Arsenic is solid in pure form and can change its oxidation state depending on the pH of the medium (Yamani et al., 2016). Due to abundance in earth crust, drinking water is mostly contaminated by arsenic which results in numerous diseases. Advanced studies have shown that the use of arsenic containing water for long duration can cause some serious diseases like cancer of skin, lungs, liver, kidney and bladder (Mahmood et al., 2014; Singh and Pant, 2004). At least 70 countries have been listed where about 140 million people are using ground water contaminated with naturally occurring arsenate anions.

Similarly chromium is also one of the most wide spread and mostly focused water contaminant due to its severe threats to human and other aquatic lives (Mustafa et al., 2010; Zelmanov and Semiat, 2011). Chromium is well-known for contaminating aqueous environment due to its extensive use in manufacturing of steel, electroplating, maintenance of wood, leather processing, production of chromic oxide and finishing of metal (Anirudhan et al., 2009; Suksabye et al., 2007). Generally, chromium is present in two oxidation state of Cr (III) and Cr (VI) with diverse toxic behavior and mobility in aqueous medium. Cr (III) has been found useful and essential component of human diet in trace amount while Cr (VI) is very harmful and have human carcinogenic effects (Bingol et al., 2009; Rojas et al., 2005). Similarly, Chromate (VI) has high water solubility and can easily transport into ground water to contaminate drinking water. Cr (VI) is adsorbed by lungs, gastrointestinal tract and even by skin. They have also the capacity to enter cell and generate intermediates that can attack DNA, proteins, membrane lipid and even cellular activities (Oliveira et al., 2010).

Due to toxicity of dichromate and arsenate anions in drinking water and the threat it poses to human health, a lot of efforts have been made to make drinking water free from Cr (VI) and As (V). For this purpose, different techniques such as filtration, chromatography, coagulation and adsorption are used to eliminate Cr (VI) and As (V) from drinking water (Li et al., 2010a; Yoon et al., 2009). However, the most frequently used technique is adsorption in which toxic material are removed from water by using zeolite, inorganic clays, alumina and titanium oxide as adsorbent (Guan et al., 2012; Jiang et al., 2015). Unfortunately, the above mentioned adsorption techniques are costly with low selectivity and percent removal. Therefore, a lot of attention is given to microgels as adsorbent of metal ions from water as microgels are efficient adsorbent in comparison to all other adsorption techniques (ur Rehman et al., 2016). Microgels are insoluble in water due to crosslinked nature of the hydrophilic functional groups (Kalshetti et al., 2012). Moreover, polymer microgels are eco-friendly, biocompatible and can be made magnetic field responsive by introducing magnetic nanoparticles into microgels network that can allow the removal of adsorbent (microgels) by externally

applied magnetic field from water after adsorption (ur Rehman et al., 2016).

In this work, p(TAEA-co-GDE) microgels are used for as adsorbent for the removal of toxic metal ions, Cr (VI) and As (V) from different water environments such as DI water, tap water, sea water and creek water. The adsorption efficiency of the prepared microgels was found to be high as compared to other values of adsorption methods that are reported for Cr (VI) and As (V) in literature (Ajmal et al., 2016; Alvarez-Ayuso et al., 2007; Sahiner et al., 2015b; ur Rehman et al., 2016). The p(TAEA-co-GDE) microgels were made magnetic field responsive for its easy recovery with little a decrease in adsorption capacity for both the metal ions. Furthermore, the adsorption competence of the prepared p(TAEA-co-GDE) microgels for both metal ions was almost same till the 5th reuse cycles.

## 2. Experimental

### 2.1. Chemicals

Analytical grade chemicals and ultra-purified distilled water were used in all the experimental work. Glycerol diglycidyl ether (GDE) and tris(2-aminoethyl) amine (TAEA) were purchased from Sigma-Aldrich. L- $\alpha$  Lecithin (surfactant) was purchased from across, and gasoline (organic solvent) was obtained from local fuel station. Acetone and cyclohexane, used for washing of synthesized microgels, were purchased from Merck. Similarly, salts like potassium dichromate ( $K_2Cr_2O_7$ ) and disodium arsenate heptahydrate ( $Na_2HAsO_4 \cdot 7H_2O$ ) were obtained from Sigma-Aldrich while  $FeCl_2 \cdot 4H_2O$  and  $FeCl_3 \cdot 6H_2O$  were obtained from Fluka and across, respectively. Sodium borohydride ( $NaBH_4$ , 98%) used as reducing agent, was purchased from Merck.

### 2.2. Synthesis of p(TAEA-co-GDE) microgels

The p(TAEA-co-GDE) microgels were prepared by simple microemulsion polymerization in which oxarane of epoxy in GDE monomer is opened via active hydrogen of amine in TAEA monomer as reported earlier by our group (Sahiner et al., 2015a). Briefly, 475  $\mu$ L of TAEA monomer and 100  $\mu$ L DI water were vortexed in an Eppendorf tube. The mixture was added to 30 mL L- $\alpha$  lecithin solution (0.1 M) in gasoline under constant stirring (1200 rpm) at room temperature. After 1 h, GDE monomer (500  $\mu$ L) was added drop wise to the reaction mixture and the reaction was further continued for 2 h under the same conditions. The prepared microgels were settled down to remove the supernatant and were made surfactant free by washing twice with cyclohexane. The microgels were further washed frequently with a mixture of acetone and water (9:1) and dried in oven at 50 °C for use in adsorption of metal ions.

### 2.3. Synthesis of magnetic p(TAEA-co-GDE) microgels

The prepared microgels were made magnetic field responsive by the introduction of iron nanoparticles within p(TAEA-co-GDE) microgels as already reported in literature (ur Rehman et al., 2016). Briefly, 0.43 g  $FeCl_2$  and 1.168 g  $FeCl_3$  (1:2 mol ratio) were sonicated in 50 mL ethanol for 30 min to prepare a homogeneous mixture of the dissolved salts. The dissolved metal ions of iron were adsorbed into p(TAEA-co-GDE) microgels by putting 0.05 g dried microgels into the metal ion solution under stirring (500 rpm) for 2 h. The metal ion loaded microgels were filtered and washed twice with ethanol to remove the loosely or unbound iron metal ions. The iron metal ions were reduced by putting the loaded microgels into 50 mL of freshly prepared 0.2 M  $NaBH_4$

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