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In this study, Fe-Al binary oxide was synthesized and evaluated for arsenic removal. Due to its large surface area, Fe-Al mixed oxide shows four times higher As(V) and As(III) adsorption capacity than conventional iron oxide. For a comparative study, single metal oxides such as iron oxide and aluminum oxide are also synthesized. The physical and chemical characteristics of the prepared adsorbents are analyzed by SEM, XRD, and BET analyzer. Through the adsorption isotherm and pH effect experiments, it is discovered that Fe-Al binary oxide shows excellent arsenic adsorption capacity compared with single metal oxides.

Keywords adsorption; arsenic; iron-aluminum binary oxide; metal oxide; pH effect

INTRODUCTION

Arsenic has been a serious concern due to its toxicity and carcinogenicity even at low concentration. Arsenic ingestion causes cancer, vascular disease, and skin alteration. The U.S. Environmental Protection Agency (USEPA) and the World Health Organization (WHO) regulate the maximum contamination level of arsenic at below 10 μg/L. There are two different inorganic redox states of arsenic, with As(V) predominant in oxidizing conditions and As(III) in reducing environments (1). These inorganic arsenic species form oxyanion in water. In neutral pH, As(V) exists as a mono and divalent anion in the water. On the other hand, As(III) forms a neutral oxyanion mainly and monovalent oxyanion marginally. Because their charges change according to pH, these compounds exhibit different mobility and adsorption properties with pH (2). Between these two inorganic arsenic species, As(III) is much more toxic, soluble and mobile than the other.

To remove such inorganic arsenic from water, several methods have been investigated, such as ion exchange (3,4), co-precipitation (5), and adsorptio (6,7). Among these methods, adsorption is considered to be the most promising technology because it is simple in operation and cost effective. Numerous studies have been done to synthesize or develop the adsorbents for arsenic removal. Metal oxide has great potential as an arsenic adsorbent (8). Streat and his coworkers removed both As(V) and As(III) using granular ferric oxide in the batch and column test and they determined the complexation mechanism between arsenic and ferric oxide (9). Yuji Arai investigated adsorption complex formation between arsenic and aluminum oxide (10). Titanium oxide was also used as an arsenic adsorbent in Bang's research (11). Especially, iron oxide (ferric oxide) shows remarkably good arsenic adsorption capacity in a wide pH range. Several modification methods have been investigated to enhance the arsenic adsorption capacity of iron oxide. Examples of these methods are the use of nano size iron oxide (12), iron oxide impregnated polymeric adsorbent (13,14), sand (15), biomass (16), zeolite (17), and activated carbon (18). Incorporation of other metal oxides with iron oxide also changed the physical properties such as the surface area, surface charge, porosity, and crytallinity (19–21). These changes might enhance the catalytic activity as well as the adsorption capacity of iron oxide.

In this study, aluminum oxide, that exhibited an extremely large surface area, was mixed with iron oxide to enhance the arsenic contact with iron oxide. Synthesized Fe-Al binary oxide was characterized. Also, the enhanced arsenic adsorption capacity of Fe-Al binary oxide compared with single metal oxide was proved through adsorption isotherm and pH effect experiments.

MATERIALS AND METHODS

Synthesis of Metal Oxide

Fe-Al binary oxide was synthesized by the sol-gel hydrothermal method. 1 mol/L of Iron (III) chloride ($FeCl_3$, Aldrich) and aluminum chloride ($AlCl_3$, Aldrich) solution

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was slowly mixed. Under vigorous magnetic-stirring, 5 mol/L of NaOH (Junsei) solution was added dropwise to the mixture. After the reaction, a gel-like brown precipitate formed and was aged for a while to settle the Fe-Al hydrogel at the bottom of the beaker. The supernatant was removed. To remove the dirt and byproducts, Fe-Al hydrogel was washed with distilled water several times and hydrothermally treated in an autoclave at 120°C for three hours. The prepared Fe-Al binary oxide was dried at 100°C. The dry material was crushed and strained though a 100 µm sieve. After that, the material was stored and sealed in a bottle for use. The synthetic reaction equation of Fe-Al binary oxide is as follows:

$$FeCl_3 + AlCl_3 + 6NaOH \rightarrow Fe(OH)_3Al(OH)_3 + 6NaCl$$

The same preparation method was used for the synthesis of single metal oxides.

Adsorbent Characterization

The configuration and morphology of the synthesized particles and the elements distributed on the surface of the mixed oxide were examined by scanning electron microscopy (SEM) with an EDAX (Hitechi, S-4800). To confirm the surface area of the synthesized adsorbent, the surface area was measured by the nitrogen adsorption method with a BET analyzer (Tristar 3000). To confirm the crytallinity of the metal oxide, X-ray diffraction analysis was carried out by D/MAX-RB (Rigaku Corporation).

Adsorption and pH Effect Test

The As(III) and As(V) stock solutions were prepared using $NaAsO_2$ and $NaHAsO_4 \cdot 7H_2O$, respectively. In all experiments, concentrated As(V) and As(III) solutions were diluted to the desired concentration. All of the experiments were carried out in 20 ml vials and 200 mg/L of adsorbent was used. The reaction temperature was kept constant at 25°C. After reaction, the vials were left for twelve hours to allow the adsorbent to settle. Concentration of arsenic in the supernatant of the solution was measured. For adsorption isotherm experiments, arsenic concentration was varied from 0.2 to 250 mg/L, the initial pH of the solution was around 6, and the pH was not controlled during the adsorption reaction. To show the effect of pH on the arsenic adsorption in the Fe-Al binary oxide, 1 N, 0.1 N HCl and 0.1 N concentration of NaOH (Thermoorion) were used.

Analytical Methods

Concentration of arsenic was analyzed with an inductively coupled plasma-optical emission spectrophotometer (ICP-OES, 730-WA, Varian Inc., USA) and an Inductively Coupled Plasma Mass Spectrometer (ICP-MS, ELAN 6000, Perkin-Elmer, USA). The detection limit of ICP-AES is approximately 50 µg/L. Arsenic concentrations lower than 50 µg/L were determined using an ICP-MS. The adsorbed mass of arsenic on the adsorbent was calculated with the following equation

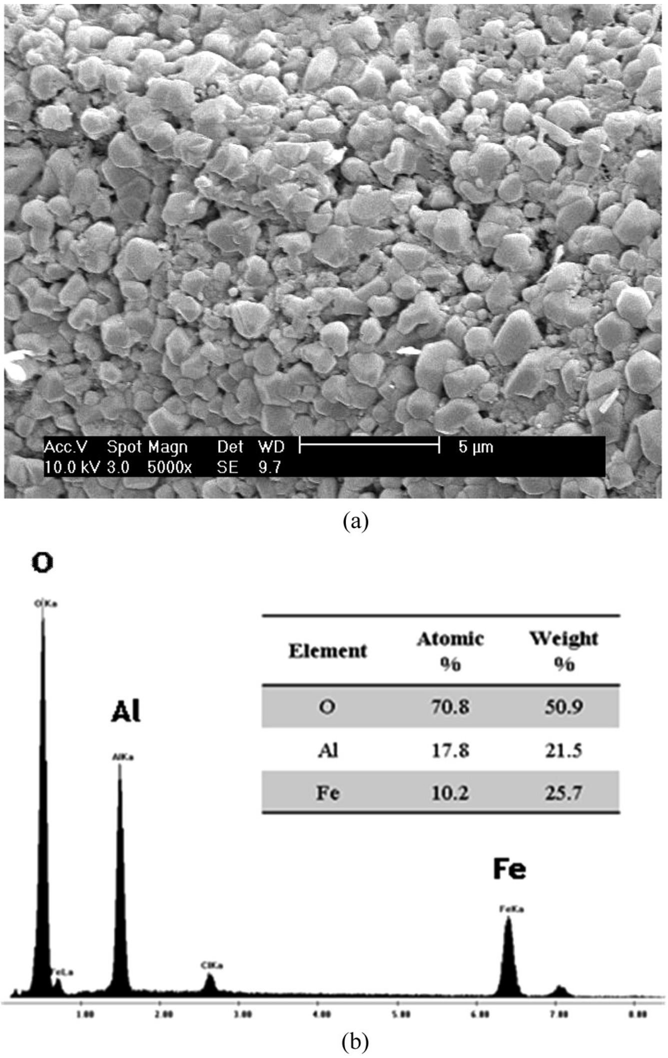
$$q = \frac{(C_{initial} - C_{final})}{m} \times V$$

where q indicates adsorbed mass of arsenic on adsorbent (mg/g) and $C_{initial}$ and C_{final} are initial and final concentration of arsenic (mg/L), respectively. V is the volume of the solution (L) and m is the mass of the adsorbent (g).

RESULTS AND DISCUSSIONS

Characteristics of Fe-Al Binary Oxide

Surface element distribution and the morphology of Fe-Al($Fe_2O_3Al_2O_3$ H_2O) binary oxide were characterized by SEM combined with EDAX. (Fig. 1) Fe-Al binary oxide



Element	Atomic %	Weight %
O	70.8	50.9
Al	17.8	21.5
Fe	10.2	25.7

FIG. 1. SEM image (a) and EDAX surface analysis (b) of synthesized Fe-Al binary oxide.

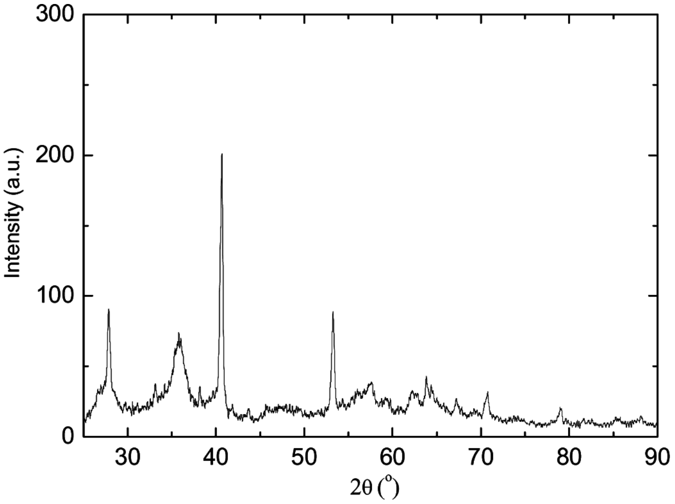


FIG. 2. X-ray diffraction patterns for Fe-Al binary oxide.

forms a porous structure that is composed of many aggregates. Synthesized Fe-Al binary oxide exhibits a large surface area (87.4 m^2/g). Considering that the iron oxide surface area is 34.3 m^2/g, aluminum oxide blending may expand the adsorbent surface area. Aluminum oxide that is synthesized by the same method shows 218.5 m^2/g of surface area.

Seventy-one percent of the Fe-Al oxide surface is covered with oxygen, and a little more Al than Fe is distributed on the surface. This result is consistent with Chubar's research (22) which found a model cluster of Fe-Al binary oxide using HyperChem7 that shows a theoretical model of the chemical structure based on quantum-chemistry. According to this model, charge distribution occurs on the adsorbent due to the different electron affinity of each element. Among Fe, Al, and O, Al and O show stronger electron donor properties than does iron. The theoretical value of the positive charge on Al is equal to $+e$. This kind of electron distribution might change the electrical properties of the Fe-Al binary oxide.

Iron oxide and aluminum oxide synthesized under the same conditions form goethite ($Fe^{3+}O(OH)$) and bohemite ($AlO(OH)$), respectively. XRD patterns obtained from the synthesized Fe-Al oxide have several peaks that indicate the slightly crystalline structure (Fig. 2). Among these, peaks shown at diffraction angle (2θ) values of 27.9, 40.8, 53.6, and 70.5 are iron oxide hydroxide (FeOOH) structures, those at 33.5, 35.8, 56.1, and 62.4 are resulted from the bayerite ($Al(OH)_3$). These results show that the synthesized adsorbent forms a mixed structure of iron oxide hydroxide and bayerite. Hydrothermal treatment might enhance the hardness of the synthesized adsorbent [23]. The characteristics of Fe-Al binary oxide and single metal oxides are described in Table 1.

Also, the point zero charge of the synthesized Fe-Al binary oxide was measured (Fig. 3). With the increase of pH, the charge on the Fe-Al binary oxide decreased. Around pH 7.2, the zeta potential of the Fe-Al binary oxide was reduced to a negative value. This means that the Fe-Al binary oxide exhibits a positive value in the natural water whose pH is approximately 5~6.

Arsenic Adsorption Isotherm on Fe-Al Binary Oxide

The adsorption capacities of the Fe-Al binary oxide for As(V) and As(III) were evaluated using the isotherms presented in Fig. 4. Fe-Al binary oxide shows four times higher arsenic (As(V) and As(III)) adsorption capacities than the other single metal oxides. Due to the large surface area, the Fe-Al binary oxide exhibits excellent arsenic adsorption capacity. Iron oxide is mixed with aluminum oxide that exhibits an extremely large surface area and it may enhance the arsenic contact with iron oxide. Also, aluminum oxide is able to adsorb arsenic although its capacity is lower than that of iron oxide. So aluminum oxide plays the role of arsenic sorbent as well as that of a supporting material for the enhancement of iron oxide contact with arsenic on the Fe-Al binary oxide. Between the two single metal oxides, aluminum oxide is less effective than iron oxide for arsenic adsorption because it is more pH sensitive and its effective pH is limited to the acidic range (3~6) (24). However, iron oxide and aluminum oxide show similar adsorption capacity in this study. Since aluminum oxide shows about six times larger surface area than iron oxide does, the actual difference in arsenic adsorption capacity between iron oxide and aluminum oxide is significant.

TABLE 1
Characteristics of Fe-Al binary oxide and single metal oxides

	Fe-Al binary oxide	Fe oxide	Al oxide
BET surface area (m^2/g)	87.42	34.32	218.46
Structure	Iron oxide hydroxide ($FeO(OH)$), Bayerite ($Al(OH)_3$)	Goethite ($Fe^{3+}OOH$)	Bohemite ($AlO(OH)$)

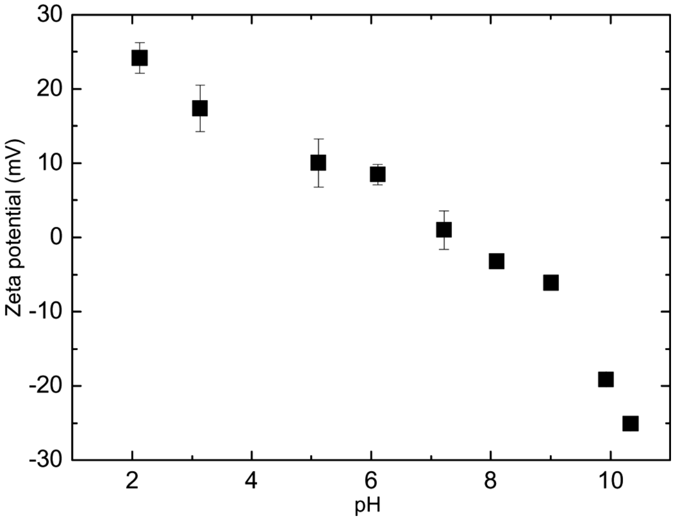


FIG. 3. Zeta potential change of Fe-Al binary oxide with pH change.

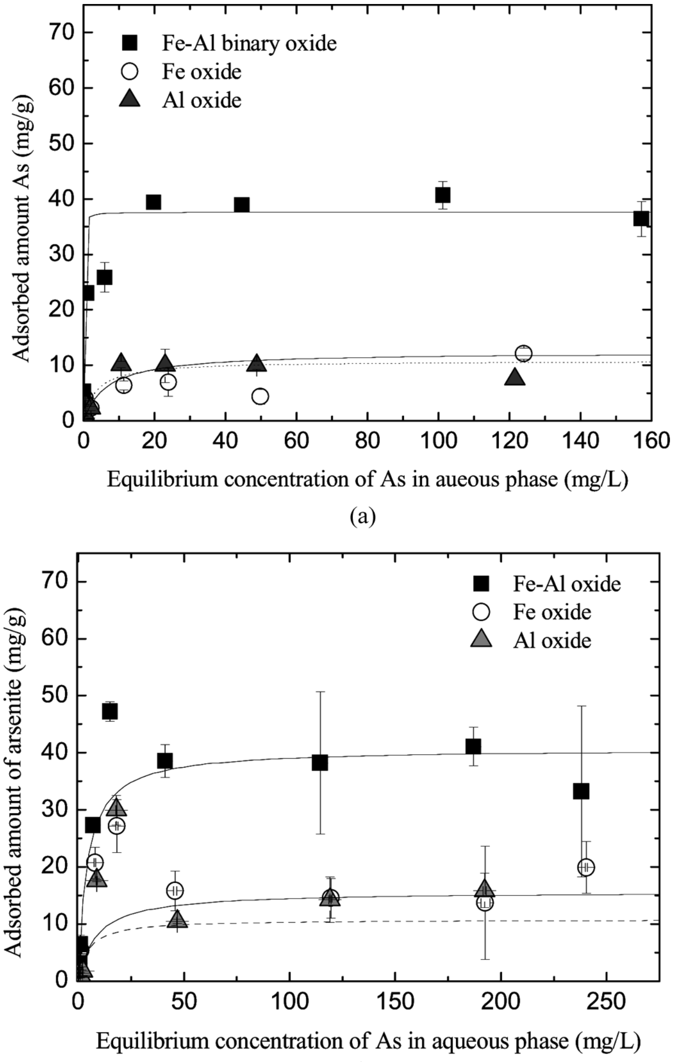
Generally, metal oxide adsorbs As(V) better than As(III) because As(V) exhibits better adsorption strength with metal oxide between two species (10,25). However, the same amounts of As(V) and As(III) are adsorbed on all metal oxides in this experiment. Because there are no other ions that can compete for adsorption on metal oxide, the excess amount of As(III) was adsorbed. So As(III) seems to show high adsorption efficiency, the same as As(V). In the arsenic desorption experiment, it was proved that As(V) is more strongly combined with Fe-Al binary oxide than As(III) is. Approximately 20% of As(III) was desorbed by contacting 0.1 M KOH or NaOH whereas only 5% of As(V) was desorbed (data not shown).

Two adsorption isotherm models, Langmuir and double scheme Langmuir isotherms, are employed to describe the adsorption isotherms.

$$\text{Langmuir isotherm } q = \frac{bkC_e}{1 + kC_e}$$

where C_e denotes the equilibrium concentration of arsenic (mg/L), k denotes the Langmuir isotherm constant (L/mg), and b indicates the maximum adsorbed amount of arsenic (mg/g). The double scheme Langmuir isotherm assumes that two different mechanisms contribute to the adsorption of the adsorbate. Because Fe-Al binary oxide contains both aluminum oxide and iron oxide, which have different functional groups that can remove arsenic, the double scheme Langmuir isotherm is also applied for the experimental data.

$$\text{Double scheme Langmuir isotherm}$$
$$q = \frac{b_1 k_1 C_e}{1 + k_1 C_e} + \frac{b_2 k_2 C_e}{1 + k_2 C_e}$$



(a)

(b)

FIG. 4. Adsorption isotherms of (a) As(V) and (b) As(III) on metal oxides.

where k_1 and k_2 are the Langmuir isotherm constant (L/mg) for different arsenic adsorption mechanisms and b_1 and b_2 is the maximum adsorbed mass (mg/g) of arsenic by different binding sites on the Fe-Al oxide. So the maximum arsenic adsorption capacity of arsenic on Fe-Al binary oxide is summarized as the values b_1 and b_2. Adsorption of As(V) and As(III) on all metal oxides fits Langmuir isotherms to a better degree than it fits the double scheme Langmuir isotherms (Table 2). However, experimental data also shows almost 1 for the R^2 value with double scheme Langmuir model. For both As(V) and As(III) adsorption, b_1 and b_2 derived from the double scheme Langmuir model, show similar values. This similarity means that the two different binding sites on the Fe-Al binary oxide (one belongs to iron oxide and the other belongs to aluminum oxide) do not show significant

TABLE 2
Model isotherm constants and linear regression coefficient of As(V) and As(III) on metal oxides

	Constant and linear regression coefficient	As(V)			As(III)		
		Fe-Al oxide	Fe oxide	Al oxide	Fe-Al oxide	Fe oxide	Al oxide
Langmuir isotherm	k (L/mg)	26.60	0.15	0.28	0.24	0.21	0.12
	b (mg/g)	37.59	12.38	10.8	40.65	10.74	15.63
	R^2	1	0.97	0.94	0.99	0.97	0.97
Double scheme	k_1 (L/mg)	30.21	–	–	0.15	–	–
	b_1 (mg/g)	20.16	–	–	20.70	–	–
Langmuir isotherm	k_2 (L/mg)	0.15	–	–	0.15	–	–
	b_2 (mg/g)	20.58	–	–	21.19	–	–
	R^2	0.98	–	–	0.98	–	–

difference in arsenic adsorption. So, the double scheme Langmuir isotherm shows similar trends with the traditional Langmuir model, which assumed mono-site arsenic adsorption.

Based on the theoretical determination of the Langmuir isotherm, both As(V) and As(III) form a monolayer on all metal oxides at the equilibrium state and the maximum adsorption capacities of the adsorbents were calculated by the Langmuir isotherm constant (*b*). The Langmuir adsorption constant (*k*) determined the slope of the isotherm. The high *k* value indicates that rapid adsorption of arsenic occurred at low equilibrium concentration. The As(V) Langmuir adsorption constant on the Fe-Al binary oxide is 26.6, which is quite different from that of As(III) (<1). This difference means that the As(V) adsorption isotherm on the Fe-Al binary oxide quickly arrived at the maximum adsorbed amount (equilibrium) even at low concentration. So, more As(V) than As(III) is adsorbed on the Fe-Al binary oxide when the initial concentration of arsenic is low.

Although the adsorption constant (*k*) is devoid of any physical-chemical meaning with respect to the forces involved in the sorption process, from this value the adsorption affinity can be predicted by the dimensionless equilibrium parameter (*r*), which is determined by the following equation (26).

$$r = \frac{1}{1 + kC_0}$$

Where k is the Langmuir adsorption constant, and C_0 is initial concentration of anionic contaminants. If the value r is lower than 1, adsorption occurs favorably. When r is greater than 1, it means that adsorption is unfavorable. Because the k values of As(V) and As(III) adsorption on Fe-Al binary oxide are positive (26.6 for As(V) and 0.24 for As(III)), their r values are lower than 1. So it is found that the adsorption of arsenic favorably occurs on the Fe-Al binary oxide. Increase of initial As(V) and As(III) concentration may lower the r value. Although both arsenic species show positive values, the As(III) adsorption constant is just slightly higher than 0. So, the r values of As(V) and As(III) differ greatly. When the initial concentration of arsenic is 5 mg/L, r values of As(V) and As(III) on Fe-Al binary oxide are 0.0074 and 0.45, respectively. From this result, it can be concluded that both As(V) and As(III) adsorption are favored on the Fe-Al binary oxide, but that As(V) adsorption is preferred to As(III) on Fe-Al binary oxide.

Effect of pH on As Adsorption on Fe-Al Binary Oxide

In this section, the pH effect on the adsorption of arsenic on Fe-Al binary oxide was investigated. Figure 5 shows the adsorbed amount of As(V) and As(III) on iron oxide, aluminum oxide, and Fe-Al binary oxide at various levels of pH. Adsorption of As(V) and As(III) on metal oxides strongly depends on the pH of the solution.

Electrostatic attraction and repulsion between the adsorbate and the adsorbent are closely related to the adsorption capacity, although the electrostatic force was not major adsorption mechanism. In this experiment, the pH of the solution not only affects the charges on the metal oxide, but also the ionization of the arsenic species. So, the adsorption of As(V) and As(III) on the metal oxides strongly depends on the pH of the solution.

At pH 2, the adsorbed mass of As(V) on Fe-Al binary oxide is approximately 22.5 mg/g. The adsorption capacity increased to 25 mg/g with the pH increase to 3∼4. The pH change from 2 to 3 results in ionization of arsenate (As(V)), negatively charged arsenate anion ($H_2AsO_4^-, HAsO_4^{2-}$) is easily adsorbed by positive Fe-Al binary oxide compared to the neutral form (H_3AsO_4). So, the increase of the pH from 2 to 3 leads to an increase of the As(V) adsorption. However, the increase of the pH not only enhances the electrical charge (valence) of the arsenate anion but also

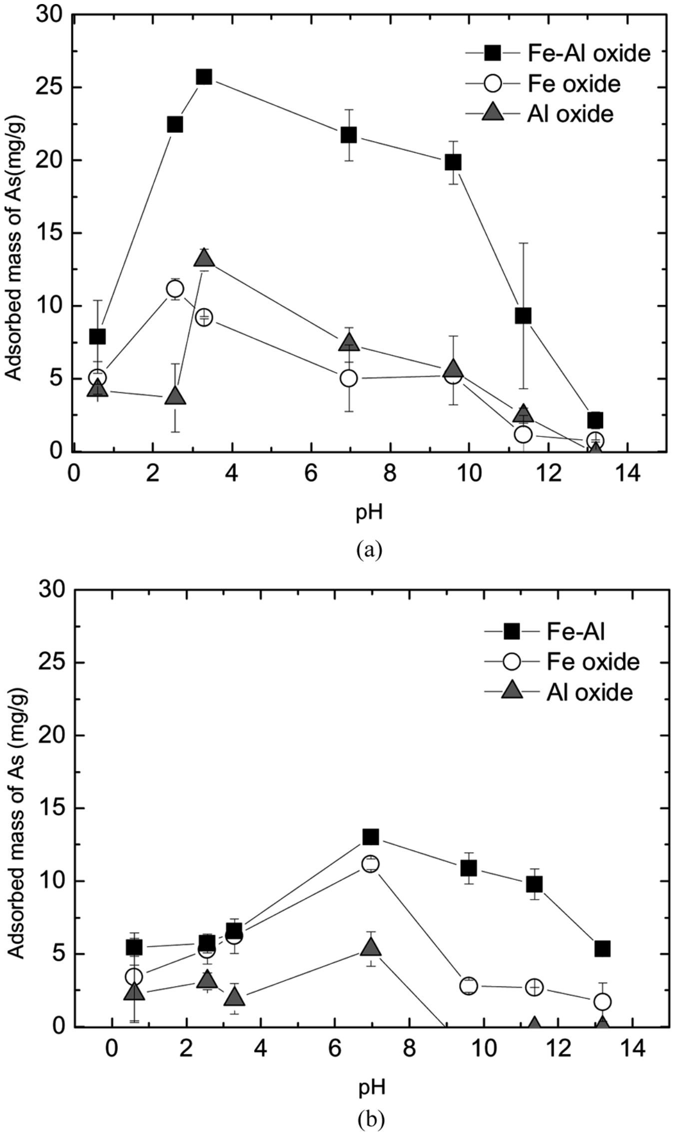


FIG. 5. Effect of pH on adsorption of (a) As(V) and (b) As(III) on metal oxides.

reduces the zeta potential of the metal oxides (Fig. 2). At pH 6, Fe-Al binary oxide exhibits positive potential but shows a reduction of about 25 mV of its zeta potential with the increase of pH from 2 to 6. So, the electrostatic attractive force between As(V) anion and Fe-Al binary oxide decreases and the adsorption of As(V) decreases.

Even at pH higher than 7, Fe-Al binary oxide shows negative values and repulsion occurs between As(V) anion and metal oxide. Also, the increase of OH^- ions interrupts the adsorption of As(V) at high pH. OH^- ions compete with arsenate oxyanions to take adsorption sites. So the highest As(V) adsorption on Fe-Al binary oxide was obtained at pH 3∼4. Iron oxide and aluminum oxide show arsenic adsorption behavior similar to that of Fe-Al binary oxide in a pH range of 3∼12. Fe-Al binary oxide shows significantly high adsorption capacity compared with that of single metal oxide over all levels of pH except for pH 2 and 12.

The adsorbed amounts of As(III) on all metal oxides are much lower than those of As(V) for all pH. The maximum adsorbed amounts of As(III) on Fe-Al binary, iron, and aluminum oxide were 13.0, 11.2 and 5.3 mg/g respectively at pH 7.

Adsorption of As(III) on all metal oxides increases up to pH 6.9 and then begins to decrease at pH levels higher than 7. Because As(III) is ionized at higher pH (9.2 and 12.7), arsenite exists in solution mainly as H_2AsO_3 and marginally as $H_1AsO_3^-$ at neutral pH (25). The amount of $H_1AsO_3^-$ increases with pH and consequently the adsorption of As(III) improves. However, the increase of pH lowers the surface charge of the adsorbent. This leads to the decrease of As(III) adsorption on all metal oxides at pH levels higher than 7. Especially, As(III) adsorption on single metal oxides drops dramatically with pH. Fe-Al binary oxide also exhibits negative potential pH levels higher than 7, like other single metal oxides and electrostatic repulsion occurs between As(III) anion and Fe-Al binary oxide. However, due to its large surface area, the Fe-Al binary oxide has a much higher chance to come in contact with As(III) compared with single metal oxide.

So, Fe-Al binary oxide shows better adsorption capacity than the other adsorbents at pH levels of 9∼11.5. However, the adsorption of As(III) on Fe-Al binary oxide and single metal oxide do not show remarkable differences in acidic pH. It seems that As(III) is ionized under strong basic conditions, which make it hard to form an anion, and so the electrical property of the adsorbent does not considerably affect the adsorption of the As(III) in acidic conditions.

CONCLUSION

In this study, Fe-Al binary oxide is synthesized and evaluated for arsenic removal. Two inorganic arsenic species (*ca.* As(V) and As(III)) are removed. Fe-Al binary oxide exhibits a larger surface area because of the blending with aluminum oxide, which shows an extremely large surface area. Synthesized Fe-Al binary oxide exhibits a slightly crystalline structure and exhibits a positive charge at neutral pH. Fe-Al binary oxide can adsorb 37.6 and 40.6 mg/g of As(V) and As(III), respectively, at neutral pH. These values are derived from the Langmuir isotherm model, which shows a high regression coefficient ($R^2 > 0.98$). Although the maximum adsorbed masses of As(V) and As(III) are similar, compared with As(III), As(V) strongly adsorbed on the Fe-Al binary oxide. The highest As(V) adsorption occurs at pH 3∼4 and Fe-Al binary oxide shows much higher adsorption capacity than single metal oxides over all levels of pH except for pH 2 and pH 12. Different from As(V), As(III) adsorption prefers pH 7 to acidic conditions.

Due to the large surface area, Fe-Al binary oxide shows higher As(III) adsorption capacity than the other single metal oxide at high pH. Approximately four times higher As(V) and As(III) adsorption was achieved with Fe-Al binary oxide than single metal oxides.

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