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Separation Science and Technology

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713708471>

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To cite this Article Zhao, Youcai , Zouboulis, A. I. and Matis, K. A.(1996) 'Removal of Molybdate and Arsenate from Aqueous Solutions by Flotation', *Separation Science and Technology*, 31: 6, 769 – 785

To link to this Article: DOI: 10.1080/01496399608001323

URL: <http://dx.doi.org/10.1080/01496399608001323>

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Removal of Molybdate and Arsenate from Aqueous Solutions by Flotation

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ABSTRACT

Ion flotation and adsorbing colloid flotation have been studied in this paper for the effective removal of molybdenum(VI) and arsenic(V) from dilute aqueous solutions. These different flotation methods were also compared. Ion flotation using a cationic surfactant (dodecylamine) as collector, as well as adsorbing colloid flotation using ferric hydroxide as coprecipitant (or sorbent) and an anionic surfactant (sodium dodecyl sulfate) as collector were examined. Laboratory-scale experiments were conducted in order to assess the effects of the following parameters on the efficiency of the process: pH value, dosages of chemical reagents, initial concentrations of arsenic and molybdenum, and the presence of foreign anions, such as Cl^- and SO_4^{2-} . In practical applications, ion flotation or adsorbing colloid flotation may be selected according to the concentration of arsenic, molybdenum, and also the initial $[\text{Mo}]/[\text{As}]$ molar ratios in solution.

INTRODUCTION

Arsenic is present usually as an admixture in molybdenum ores, such as molybdenite (MoS_2). The wastewater generated during the hydrometallurgical treatment of molybdenum minerals usually contains both arsenic(V) and molybdenum(VI) species. In addition, the impurities of arsenic in molybdenum leaching liquors are currently removed by precipitation by adding magnesium chloride. This results in a precipitate containing

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arsenic(V) and molybdenum, which should be further treated before it is environmentally safe for disposal. An alternative route for the detoxification of this precipitate is to retransfer any arsenic and molybdenum it contains back to the solution and then to remove them from the solution. In general, the content of molybdenum and arsenic in the wastewater generated during molybdenum processing or the leach solution of the resultant precipitate varies between 100–1000 and 5–40 mg/L, respectively (1). The removal of arsenic and molybdenum from waste streams is highly desirable, mainly for health and environmental reasons.

In view of the efficient operation of flotation in mineral processing and also in the treatment of industrial wastewaters (2), it seemed worthwhile to explore the possibility of using this process for the removal of molybdenum and arsenic ions from dilute solutions. Existing methods of arsenic removal involving flotation include ion flotation (denoted IF) in a neutral medium with rosin amine acetate as a surfactant (3) or by adsorbing colloid flotation (denoted ACF) at pH 4–5 with ferric hydroxide as a sorbent; various surfactants were used for the latter, such as sodium dodecyl sulfate, sodium oleate, etc. (4–8). These methods are usually used for the treatment of wastewater containing a relatively low content of arsenic or for analytical purposes. The selective separation of trace pollutants (such as arsenic) from aqueous solutions using flotation techniques has also been investigated (9, 10).

The removal and recovery of molybdenum as a salable product from lean solution, or the separation of low concentration of molybdenum from solution by IF has also been studied by using several surfactants such as cetylamine or quaternary amine (11–13).

The simultaneous removal of arsenic and molybdenum from solutions by flotation has not been reported in the literature, at least to our knowledge. In the present paper, ion flotation using dodecylamine (as collector) and, in parallel, adsorbing colloid flotation using ferric hydroxide (as co-precipitant) and sodium dodecyl sulfate (as collector) are examined systematically for the removal of arsenic and molybdenum species from an aqueous solution containing both of them.

Aquatic Chemistry of the System

Molybdenum exists in water in the +3, +4, +5, and +6 valency states. The +6 state is stable over a broad region of the potential vs pH diagram in the absence of other complexing agents (1). At above pH 7 or 8, Mo(VI) occurs as the tetrahedral monomeric molybdate ion MoO_4^{2-} , but polymerization occurs at concentrations in excess of 10^{-4} M at lower pH values. The MoO_4^{2-} ion can form insoluble precipitates with most cations, with

the exception of the alkali metal ions. For example, when Fe^{3+} , Ca^{2+} , or Pb^{2+} is introduced into an aqueous solution of molybdate, the corresponding precipitate may be formed in weak acidic or alkaline media. When the concentration in a solution of molybdenum is higher than 10^{-4} M, the main species of polymolybdates are $\text{Mo}_7\text{O}_{22}(\text{OH})_2^{4-}$ (at pH 2.5–3.5), $\text{Mo}_7\text{O}_{23}(\text{OH})^{5-}$ (pH 3.5–4.5), $\text{Mo}_7\text{O}_{24}^{6-}$ and MoO_4^{2-} (pH 4.5–6.5), and MoO_4^{2-} (pH > 6.5–7). If the pH value is lower than 3.8 and the concentration of molybdenum is lower than 10^{-4} M, the main species are H_2MoO_4 (pH < 3.5) and HMnO_4^- (pH 3.5–3.8), while MoO_4^{2-} exists at pH > 3.8 (14).

The chemistry of arsenate is much simpler (15). The main species of arsenates are H_3AsO_4 (pH < 2), H_2AsO_4^- (pH 2–6), HAsO_4^{2-} (pH 6–11), and AsO_4^{3-} (pH > 11). For a solution with a certain pH value, several arsenate ions may exist simultaneously. Most of heavy metals and ferric salts of arsenate(V) are insoluble.

When solutions of molybdate and arsenate are mixed and acidified, the species in the resulting solution is much more complex. Besides the species of molybdates and arsenates mentioned above, various molybdo-arsenic anions with different $[\text{Mo}]/[\text{As}]$ molar ratios form, depending on concentrations of molybdenum and arsenic and also the pH value. Among them, $\text{H}_3\text{AsMo}_{12}\text{O}_{40}$ and its anion is very stable in solution when the pH is less than about 3. The latter has been studied extensively and is used in one of the important colorimetric analytical methods of arsenic(V); this can be prepared easily by ether extraction from strong acidic solution. When the pH is raised, it will degrade to species with smaller $[\text{Mo}]/[\text{As}]$ molar ratios, such as $[\text{AsMo}_{11}\text{O}_{39}]^{7-}$, $[\text{As}_2\text{Mo}_{18}\text{O}_{62}]^{6-}$, $[\text{AsMo}_8\text{O}_{29}]^{5-}$, $[\text{AsMo}_6\text{O}_{23}]^{5-}$, and $[\text{As}_2\text{Mo}_6\text{O}_{26}]^{6-}$. The relationship between these species and the pH is quite complex. It has been confirmed that molybdoarsenic anions will decompose into AsO_4^{3-} and MoO_4^{2-} ions completely when the pH is higher than 10–11. At pH 6–7, $[\text{As}_2\text{Mo}_6\text{O}_{26}]^{6-}$ is stable in solution. The heteropoly- and isopolyoxometalates of molybdenum, tungsten, and vanadium have been reviewed by Pope (14).

EXPERIMENTAL

Materials

All chemicals used in this study were of analytical reagent grade. Dodecylamine (RNH_2) in ethanol solution was used as the surfactant for ion flotation, and sodium dodecyl sulfate (SDS) and ferric hydroxide (formed in-situ from ferric chloride) were used as the surfactant and coprecipitant, respectively, for adsorbing colloid flotation. Sodium salts of chloride and sulfate were used as the source of foreign anions when the ionic strength

was examined. The arsenic(V)- and molybdenum(VI)-bearing stock solutions were prepared by dissolving a predetermined amount of $\text{Na}_2\text{HAsO}_4 \cdot 7\text{H}_2\text{O}$ and $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ in deionized water.

Procedures

The removal of arsenic and molybdenum from As- and Mo-bearing feed solutions was studied by using a laboratory bench-scale dispersed-air flotation column which was constructed with a 34-mm i.d. and 330 mm long glass tubing, with a fritted glass disk as the bottom in the microcell. The disk, which had D_4 nominal porosity (i.e., 10–16 μm), was used as a sparger to generate the necessary bubbles for flotation.

In IF experiments the feed solution was prepared by mixing a given volume of As and Mo stock solutions in a beaker, diluting, and then adding ethanolic dodecylamine solution while stirring with a magnetic stirrer. The content of ethanol in the feed was always kept at 0.5% v/v, and a total volume of 100 mL solution was used for all the experiments. The feed should be mixed until the pH is stable (around 10–15 minutes), before it is introduced into the flotation microcell. To start a flotation test, a predetermined air flow rate was diverted to the unit. At the optimum conditions a white precipitate could be seen attached to the glass wall and no foam was observed to flow out from the top of the column. An air flow rate of 300 cm^3/min with a 10-minute flotation time was found to be necessary for equilibrium; if the flow rate was lower, the time required for flotation equilibrium was longer. The feed solution was originally turbid, but became clear during floating. After the necessary time the gas was turned off and an aliquot from the remaining solution was taken from the lower part of the column in order to determine the arsenic and molybdenum concentrations. Previous results showed that the addition of ethanol alone had little effect on process efficiency.

In ACF experiments the procedure was nearly the same. The feed was prepared by mixing the stock solutions of As, Mo, and Fe(III) (and foreign anions, where required), diluting as needed, and adjusting the solution pH to the desired value while stirring for about 5 minutes. Then sodium dodecyl sulfate was added and stirring was resumed for another minute. The solution was introduced into the flotation microcell with an air flow rate of 50 cm^3/min , for at least 5 minutes. At the optimum conditions, a little foam overflowed from the column and a dark brown precipitate could be seen floating on the surface of the solution. It was found that the maximum removal of ions involved could be reached in approximately 10 minutes.

The initial concentrations of arsenic(V) and molybdenum(VI) in the feed used were at 15.4 and 211.4 mg/L, unless otherwise stated. Arsenic was chemically analyzed with the hydrazine sulfate-reduced molybdenum blue colorimetric method; molybdenum was determined with the SnCl_2 -NHSCN and Cu^{2+} -NHSCN colorimetric methods for solutions with and without ferric ions (16).

RESULTS

Ion Flotation

Effect of pH Value

The removal of molybdenum(VI) and arsenic(V) depended strongly on the solution pH. It can be seen from Fig. 1 that the maximum removal of both molybdenum and arsenic was obtained in the pH range of about 2.5–4.5, which was selected for the following experiments. The percent removal (denoted as Re) of arsenic was closely related to that of molybde-

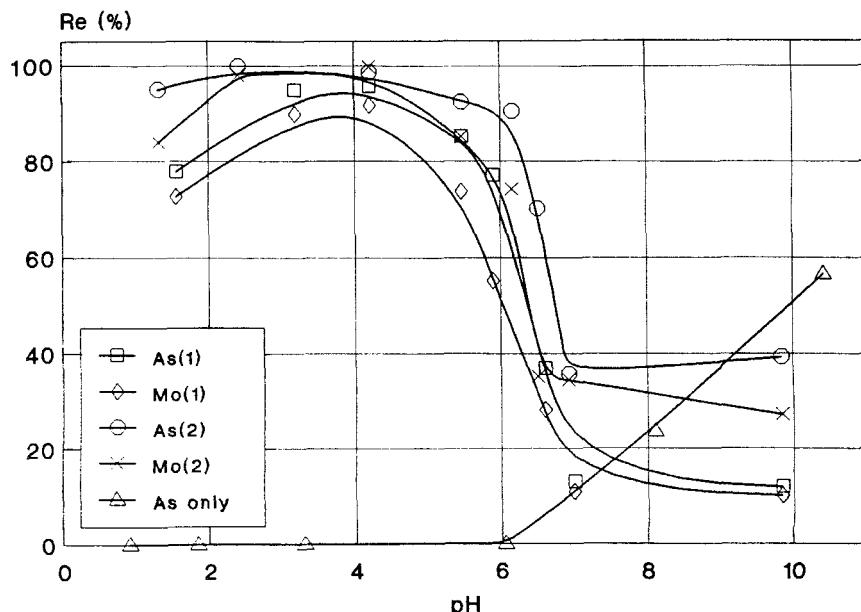


FIG. 1 Influence of solution pH on molybdenum and arsenic removal from solutions by ion flotation at different dodecylamine content: (1) 283.76, (2) 354.70 mg/L RNH_2 .

num. Arsenic could not be removed from a solution containing only As(V) at pH 2–6. The flotation behavior of molybdenum in solutions containing only molybdenum was found to be very similar to that in As–Mo systems (not shown in the figure).

Effect of Dodecylamine Addition

The removal of molybdenum and arsenic from solution was found to increase linearly with an increase of dodecylamine content. Over 99% removal of both elements could be obtained at amine contents higher than 350 mg/L, as shown in Fig. 2.

Effect of Arsenic Concentration

When the initial content of arsenic in solution was lower than 15 mg/L, over 99.4% of arsenic could be removed, while the separation of molybdenum was always quantitative, regardless of its concentration, under the conditions of Fig. 3.

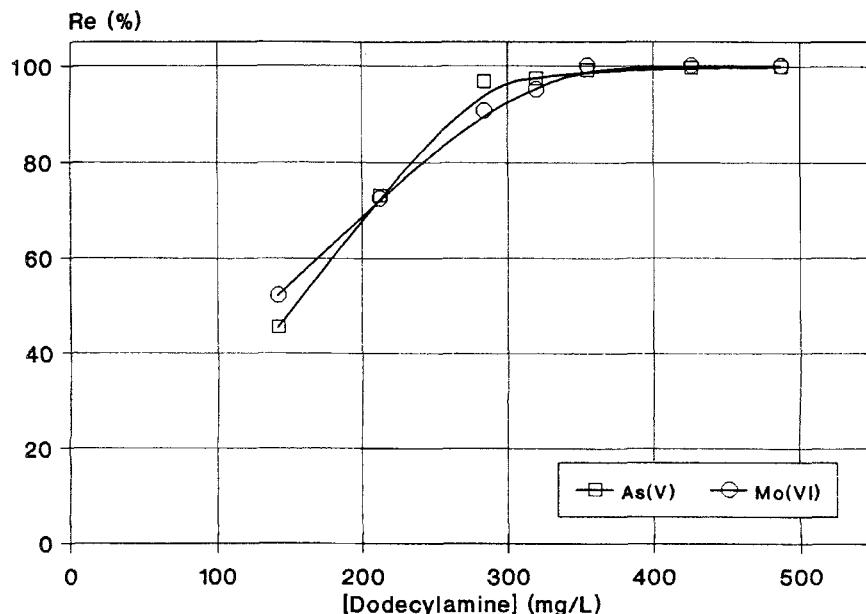


FIG. 2 Effect of dodecylamine addition during ion flotation; pH 3.6 (the other conditions are the same as in Fig. 1).

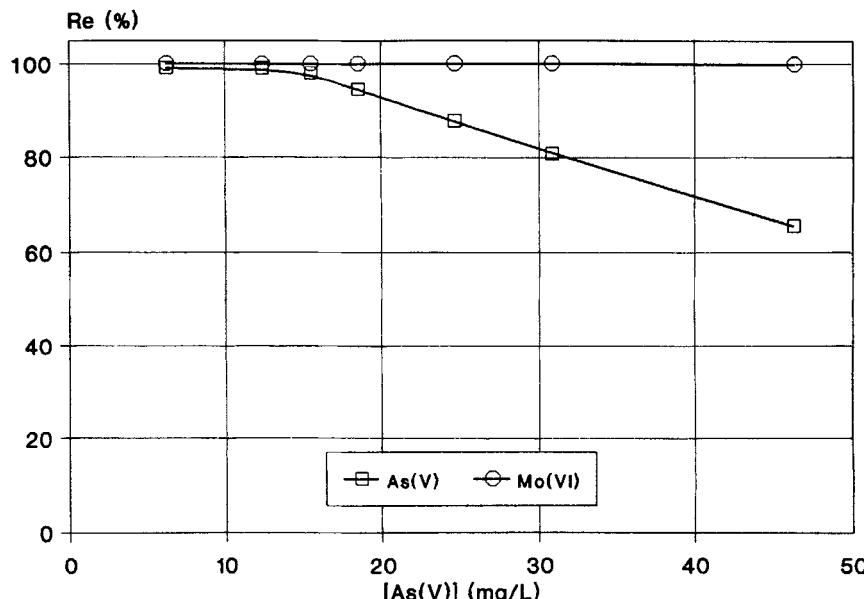


FIG. 3 Effect of arsenic(V) content in solution during ion flotation; $[RNH_2] = 354.70$ mg/L.

Effect of Molybdenum Concentration

No arsenic was removed without the presence of molybdenum in solution; it seemed that arsenic floated as molybdoarsenic anions. When the content of molybdenum was increased from 120 to 210 mg/L, its removal was found to be quantitative and then decreased linearly with any further increase of molybdenum, as shown in Fig. 4.

When the concentration of molybdenum was lower than 120 mg/L, the RNH_2 content (354.70 mg/L) was in great excess and difficulties were encountered for the effective flotation of both elements. When the RNH_2 content was used at a molar ratio of $[Mo]/[RNH_2]$ equal to 1:1, the respective results were as presented in Table 1.

It can be noticed that the separation of molybdenum from solution was always quantitative. However, the simultaneous complete removal of arsenic was obtained only when the initial $[Mo]/[As]$ molar ratio in the solution was higher than 6 if dodecylamine was present at the necessary concentration. Otherwise, i.e., when the initial $[Mo]/[As]$ molar ratio was lower than 6, quantitative removal from solution was possible for molyb-

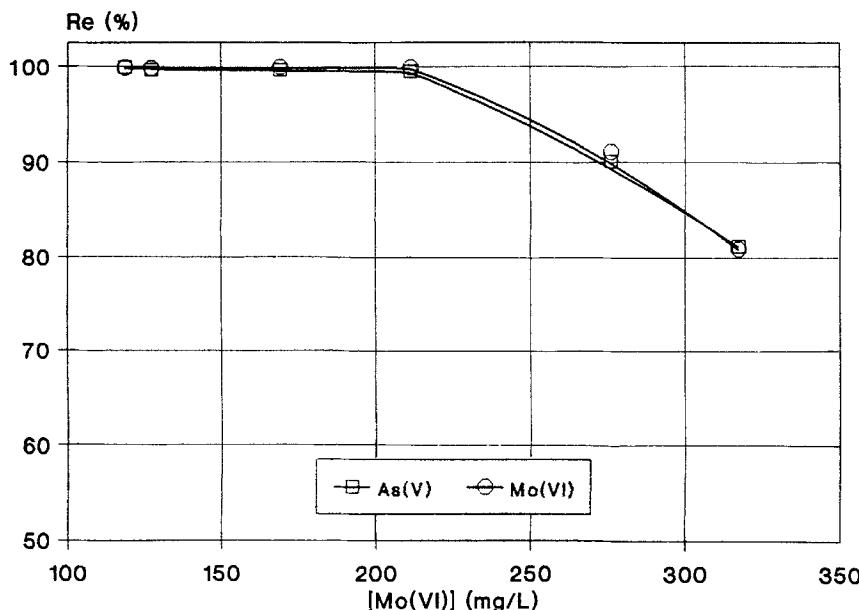


FIG. 4 Effect of initial molybdenum content in solution during ion flotation; $[As] = 15.41 \text{ mg/L}$.

TABLE I

Relationship between the Removal of As and Mo from Aqueous Solutions by Ion Flotation and the Content of Primary Amine; $[Mo]/[RNH_2]$ Molar Ratio Equal to 1:1; $[As] = 15.41 \text{ mg/L}$ (0.205 M), [Ethanol] = 0.5%, pH 3.6

	Run					
	1	2	3	4	5	6
RNH_2 (mM)	0.11	0.22	0.44	0.77	1.1	1.32
Mo (mM)	0.11	0.22	0.44	0.77	1.1	1.32
RNH_2 (mg/L)	21.04	42.96	85.92	142.72	203.89	244.67
Mo (mg/L)	10.57	21.14	42.28	73.99	105.7	126.84
Mo removal (%)	99.99	99.89	99.99	99.87	99.87	99.76
As removal (%)	12.72	18.57	37.47	53.16	71.01	99.87
Initial Mo/As molar ratios	0.55	1.1	2.2	3.85	5.5	6.60

TABLE 2

Effect of High Content of Primary Amine on the Removal of Arsenic and Molybdenum from Aqueous Solutions by Ion Flotation; $[\text{Mo(VI)}]/[\text{As(V)}]$ Molar Ratio Equal to 3.6; $[\text{Mo}] = 211.41 \text{ mg/L}$ (2.20 mM), $[\text{As}] = 46.23 \text{ mg/L}$ (0.62 mM), pH 3.6

Run	RNH_2 (mg/L)	Mo removal (%)	As removal (%)
1	354.7	99.87	65.45
2	496.17	99.89	67.73
3	637.94	99.98	72.46

denum but impossible for arsenic. An increase of dodecylamine improved the separation of arsenic a little when the $[\text{Mo/As}]$ molar ratio was kept constant at 3.6, as shown in Table 2.

Effect of Foreign Anions Presence

The addition of Cl^- ions had no effect on the efficient removal of arsenic and molybdenum by IF. However, the presence of SO_4^{2-} ions depressed the removal of both elements remarkably (see Fig. 5). It is worth noting

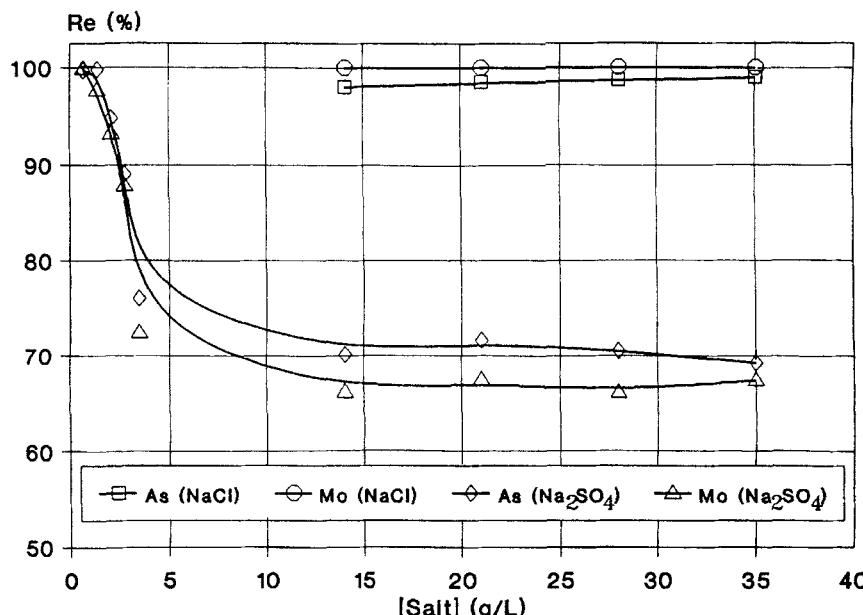


FIG. 5 Influence of foreign ions present as sodium salts during ion flotation; pH 3.3.

that in most molybdenum hydrometallurgical processes only HCl acid is used; therefore, the negative effect of SO_4^{2-} on flotation is not expected. If ion flotation is going to be applied for the treatment of leachates resulting from molybdenum solid wastes, the use of sulfuric acid must be avoided.

Adsorbing Colloid Flotation

Effect of pH

When the initial $[\text{Mo}]/[\text{As}]$ molar ratio in solution was lower than 6, it was impossible to remove arsenic completely by IF using dodecylamine as the surfactant. In this case, an alternative method should be applied.

Figure 6 shows the relationship between the removal of both arsenic and molybdenum species and the pH of the solution when using ACF with ferric hydroxide as the coprecipitant and sodium dodecyl sulfate as the collector. It can be seen that quantitative removal was obtained in the pH range 3.5–4.5, which was used for the following experiments. The results show that no (or little) flocs were formed when the solution pH was lower

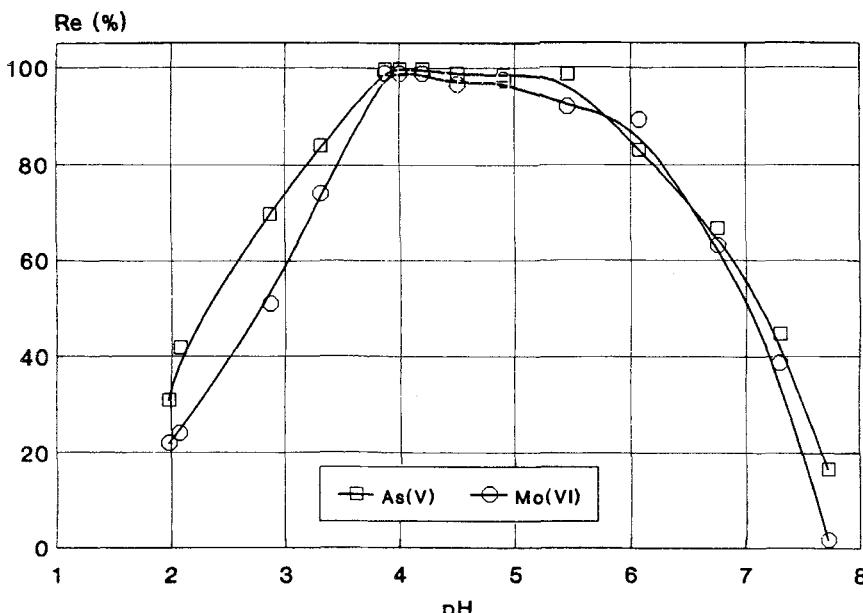


FIG. 6 Influence of solution pH on molybdenum and arsenic removal from solutions by adsorbing colloid flotation; $[\text{Mo}] = 48.00 \text{ mg/L}$, $[\text{As}] = 15.41 \text{ mg/L}$, $[\text{SDS}] = 54.13 \text{ mg/L}$, $[\text{Fe(III)}] = 134.89 \text{ mg/L}$.

than 3.5, and the flocs formed at pH values over 4.5 could be floated effectively under the given conditions. However, the unfloated flocs settled rather quickly.

Effect of SDS Addition

When the surfactant content in a solution was higher than 50 mg/L, 98% separation efficiency of arsenic and molybdenum was obtained (see Fig. 7). Nevertheless, the presence of an excessive amount of SDS led to the production of a large amount of foam, which removed a large part of the solution.

Effect of Fe(III) Addition

The removal of arsenic and molybdenum anions from an aqueous solution increased as the Fe^{3+} concentration was increased; an over 98% removal efficiency of molybdenum and arsenic was obtained when the ferric content was higher than 130 mg/L (see Fig. 8).

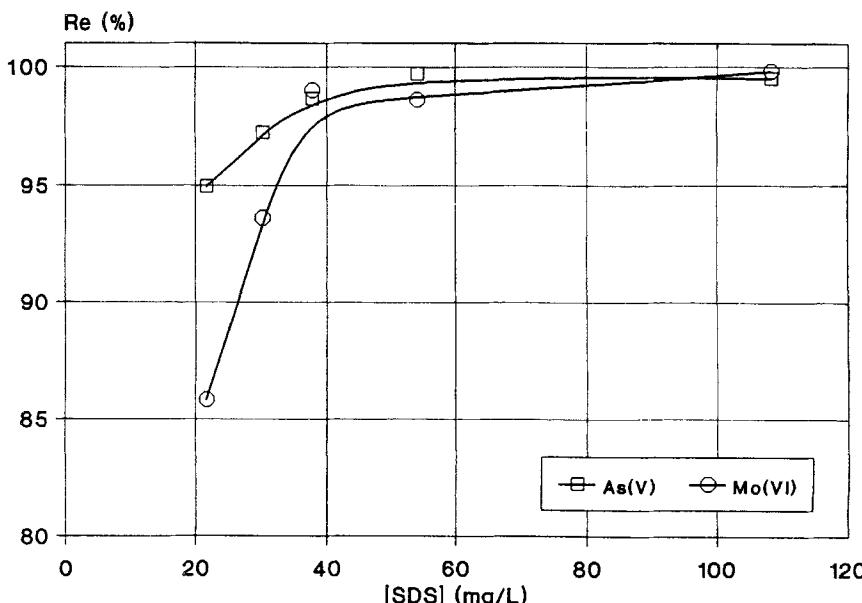


FIG. 7 Effect of sodium dodecyl sulfate addition during adsorbing colloid flotation; pH 4.0 (the other conditions are the same as in Fig. 6).

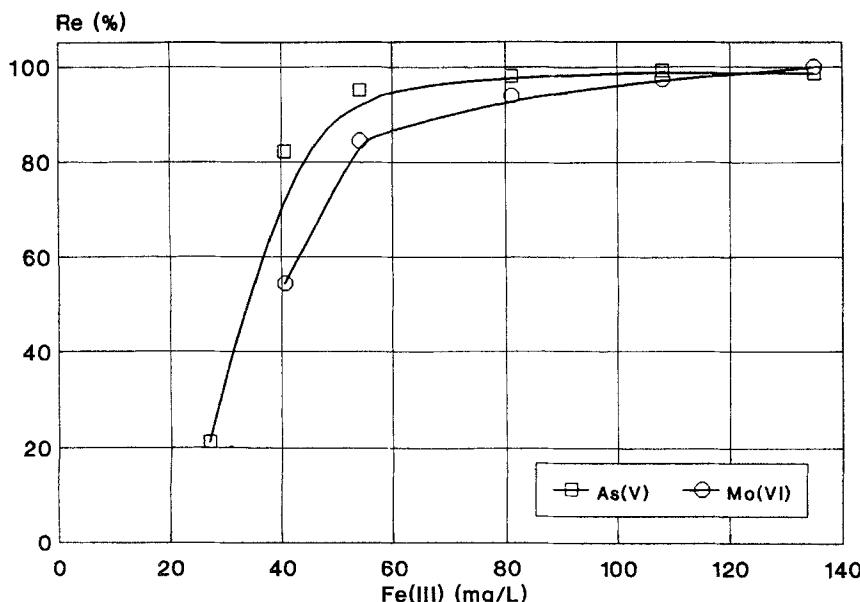


FIG. 8 Influence of ferric ions addition during adsorbing colloid flotation; $[SDS] = 54.13 \text{ mg/L}$.

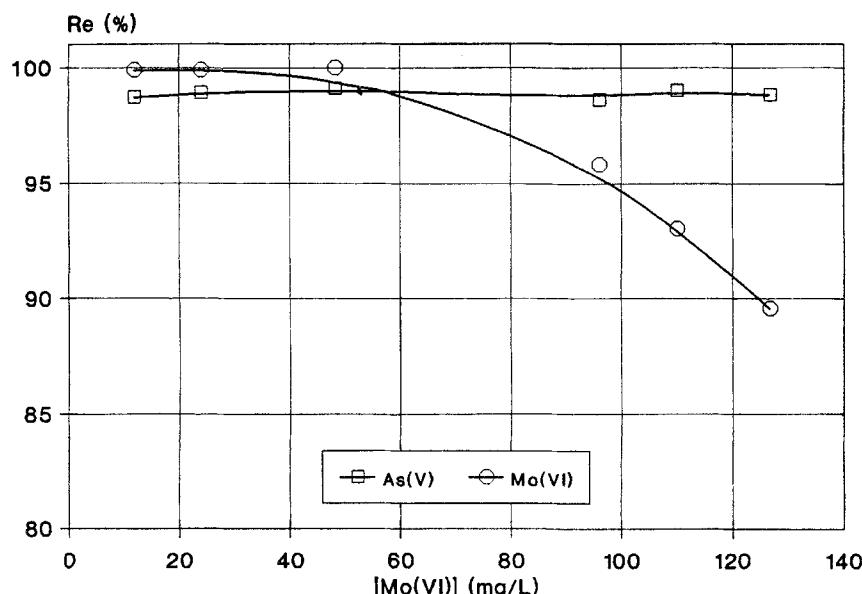


FIG. 9 Effect of molybdenum content in solution during adsorbing colloid flotation; $[Fe] = 134.89 \text{ mg/L}$.

Effect of Molybdenum Content

The removal of arsenic was always over 95% regardless of the molybdenum content in a solution. However, the quantitative removal of molybdenum could only be obtained when its initial content in a solution was lower than 60 mg/L under the conditions shown in Fig. 9. It was observed in the experiments that a white precipitate was formed, so the flotation system described is not suitable for the removal of a high molybdenum content from aqueous solutions.

Effect of Arsenic Content

When the arsenic content in a solution was lower than about 60 mg/L, over 98% removal efficiency of arsenic could be obtained, as shown in Fig. 10. A yellow precipitate appeared in the experiments when the arsenic concentration in the solution was higher than 70 mg/L, and although this precipitate settled quickly, it was nonfloatable by SDS.

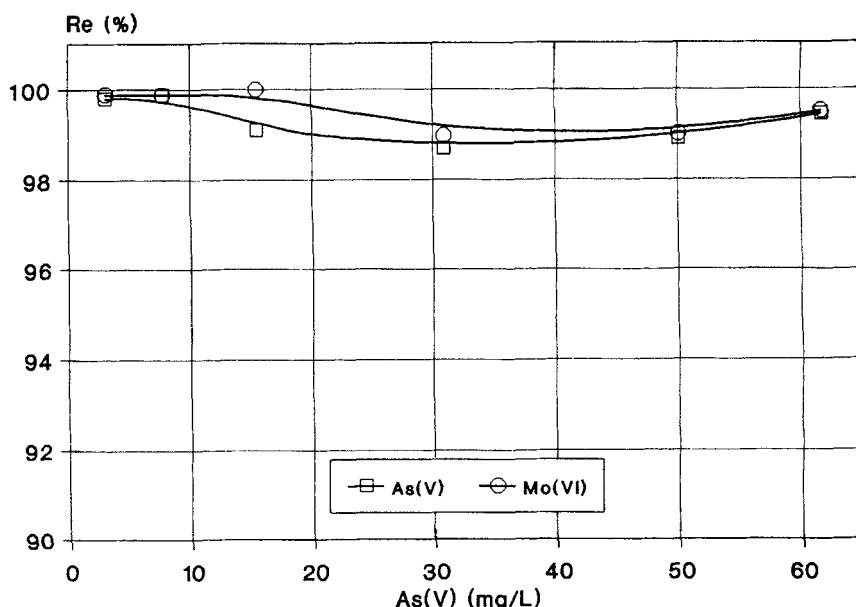


FIG. 10 Effect of arsenic content in solution during adsorbing colloid flotation; [Mo] = 48.00 mg/L.

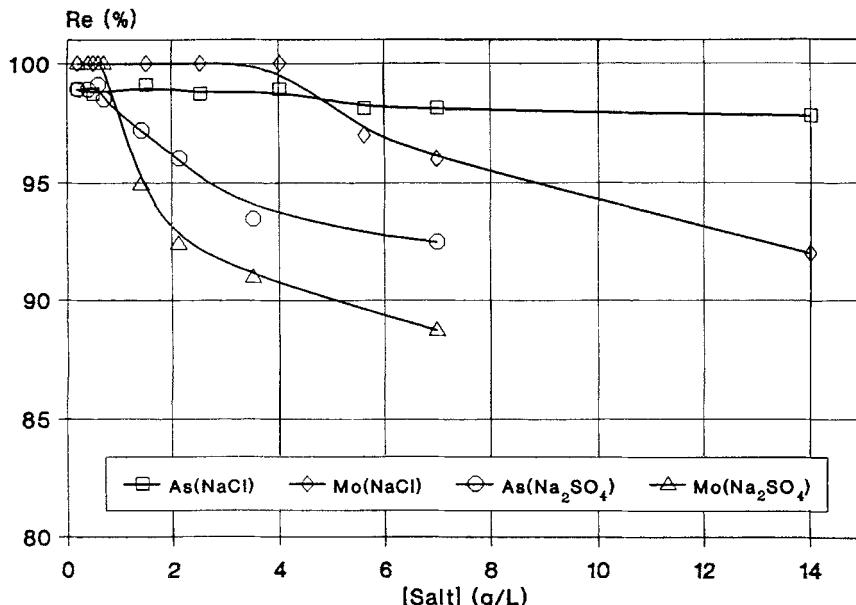


FIG. 11 Influence of foreign ions present as sodium salts during adsorbing colloid flotation.

Effect of Foreign Ions

Little effect by the addition to the solution of NaCl and Na₂SO₄ salts on flotation was noticed when the contents of these salts were lower than 4 and 0.6 g/L, respectively (see Fig. 11). However, the removals were decreased when the salt contents were further increased. Actual molybdenum hydrometallurgical wastewaters usually contain chloride ion, but little or no sulfates.

DISCUSSION

One of the advantages for the separation of arsenic and molybdenum ions from aqueous solution by IF is that only dodecylamine as a collector needs to be added. In practical applications, dodecylamine can be dissolved in hot water and then introduced into the solution used for flotation, thus avoiding the consumption of ethanol, which had relatively little effect on flotation.

Ion flotation has some disadvantages, e.g., it cannot be used for quantitative removal of arsenic from solution when the initial [Mo]/[As] molar

ratio is lower than 6, although in this case molybdenum can be removed completely, regardless of the [Mo]/[As] ratio.

In this case, ACF should be used for the complete removal of arsenic and molybdenum species from solution. This method is only suitable for the treatment of wastewaters containing arsenic and molybdenum at concentrations lower than 70 and 60 mg/L, respectively (regardless of the initial [Mo]/[As] molar ratio).

According to the results presented in this article, it seems more favorable to use ion flotation for the removal of arsenic and molybdenum species from solution when the initial [Mo]/[As] molar ratio is higher than 6. The volume of foamate formed during IF is relatively small. This is important to the further treatment of foamate and recycling of the surfactant when the recovered arsenic and molybdenum species are possibly salable products. It was also observed to be much more difficult to recycle the surfactant from the foamate during ACF.

The interaction mechanisms between Fe(OH)_3 -bearing arsenic floc and SDS have been interpreted by zeta-potential, infrared spectra analysis, and molecular orbital theory (4). The parameters influencing flotation in the removal of metal ions (such as As) were reported elsewhere (17).

When arsenic and molybdenum were removed in the present investigation from solution by ion flotation, it was found that the removal of arsenic is linearly related to that of molybdenum. It is proposed that arsenic is floated as molybdoarsenic anions. These elements may form a variety of anions with different [Mo]/[As] molar ratios in solution, such as $[\text{AsMo}_{12}\text{O}_{40}]^{3-}$, $[\text{As}_2\text{Mo}_{18}\text{O}_{62}]^{6-}$, $[\text{AsMo}_8\text{O}_{29}]^{5-}$, $[\text{AsMo}_6\text{O}_{23}]^{5-}$, $[\text{As}_2\text{Mo}_6\text{O}_{26}]^{6-}$, etc. At lower pH values the main arsenate ions seems to be H_2AsO_4^- , whose charge is less than that of the respective molybdoarsenic anions. Thus, it becomes easier for molybdoarsenic anions to form a foamate with RNH_3^+ and makes the arsenic species floatable. Recall that arsenic cannot be removed at all in the pH range between 2 and 6 without the presence of molybdenum in solution.

CONCLUSIONS

1. Both arsenic and molybdenum in aqueous solution can be effectively removed by ion flotation, using dodecylamine as the collector, when the initial [Mo]/[As] molar ratio is higher than 6, in the optimum pH range of 2.5–4.5, regardless of the initial content of arsenic and molybdenum. The foamate can be collected rather easily for further possible treatment and recycling of surfactant, as well as for recovery of the arsenic and molybdenum. The arsenate ion is considered to be floated as molybdoarsenic foamate, and its removal is directly related to that of molybdenum.

2. Adsorbing colloid flotation using ferric ions as the coprecipitant or sorbent and sodium dodecyl sulfate as the collector can be used for the effective removal of arsenic and molybdenum from aqueous solution at an optimum pH value of 3.5–4.5 when the contents of these two elements are lower than 70 and 60 mg/L, respectively, regardless of the initial [Mo]/[As] molar ratio in the solution. No direct relationship between the removal of arsenic and molybdenum from solution was observed.

3. In practical applications, the ion flotation process is preferred because of its advantages over adsorbing colloid flotation, such as the easy collection of foamate. Therefore, the applied surfactant can be recycled, and the arsenic and molybdenum can be recovered.

ACKNOWLEDGMENTS

Thanks are due to the European Union which supported one of the authors (Y.Z.) by a EU Postdoctoral Fellowship (Contract No. CI1*-CT94-0568) to carry out this research work.

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Received by editor July 14, 1995