

Assessing arsenic leachability from pulverized cement concrete produced from arsenic-laden solid CalSiCo-sludge

Puspendu Bhunia, Anjali Pal*, Manas Bandyopadhyay

Department of Civil Engineering, Indian Institute of Technology, Kharagpur 721302, India

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Abstract

Synthetically prepared arsenic-laden CalSiCo-sludge was converted to pulverized cement concrete (PCC) using solidification/stabilization technology with cement. Batch leaching experiments were conducted to estimate the leaching of As(III) and As(V) from the CalSiCo-sludge as well as from the PCC. The leaching of As(III) and As(V) was found to be the function of time, pH and concentration of anions such as Cl^- , NO_3^- , and SO_4^{2-} present in the extraction fluid. It is observed that from the CalSiCo-sludge the leaching of As(III) is >0.05 mg/l (which is above the permissible limit for arsenic in drinking water) at any pH. But in case of As(V) the leaching is >0.05 mg/l only at $\text{pH} > 8$ and at $\text{pH} < 4$. It is noted that maximum leaching occurs when the extraction liquid contains Cl^- . In contrary, NO_3^- and SO_4^{2-} have negligible effect on arsenic leaching from the CalSiCo-sludge. Extraction tests were carried out to determine the maximum leachable concentration under the chosen conditions of leaching medium and leaching time. Leaching of As(III) and As(V) from exhausted arsenic-laden CalSiCo-sludge and from PCC was carried out in both tap water and rain water. It was noticed that tap water has no effect in leaching of arsenic from CalSiCo-sludge but rain water causes significant amount of leaching, which is mostly due to pH effect. However, in all cases the leaching of As(III) was more than that of As(V). When compared with CalSiCo-sludge PCC showed negligible leaching of arsenic. It was noticed further that the variation of 28 days compressive strength was within 15% of the original strength after replacing 35% cement with exhausted CalSiCo-sludge.

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1. Introduction

The presence of arsenic in ground water and wastewater has been of great public concern. The researches on arsenic are mostly focused on the awareness building and the development of water treatment system for the removal of arsenic from drinking water. However, the disposal of arsenic sludge generated from the treatment processes is one of the issues that has to be attended by either the sponsors of the technology development and/or the researchers. It is important to address such an important issue in the present day context, and this has prompted the present investigators to undertake the work on the management of the waste sludge derived from the adsorption of arsenic.

Natural and synthetic adsorbents containing calcium, copper and silica-based materials are known for arsenic removal. A white cement-based adsorbent CalSiCo (patented trade name)

with Ca, Si, Cu as major elements present in it was developed in our laboratory and evaluated for its potential application for arsenic removal [1]. Being highly efficient and cost-effective this adsorbent is being used since last 10 years for arsenic removal in our laboratory, as well as in the fields.

It was reported that at the concentration level of 1.0 mg/l, up to ~85% of As(III) removal at $\text{pH} > 9.0$ and ~99% removal of As(V) at $\text{pH} 7.2$ was possible with an adsorbent (CalSiCo) dose of 10 and 5 g/l for As(III) and As(V), respectively. Reuse of the adsorbent for four cycles is possible with lesser efficiency of removal. But finally after a few cycles the adsorbent needs to be exposed off. Recently, solidification and stabilization techniques have emerged as one of the viable alternatives for waste disposal [2–4]. Chemical stabilization processes are currently in use to treat industrial and hazardous wastes. Chemical solidification and stabilization refer to waste treatment that results in the combined effect of improvement of physical properties (mechanical stabilization), encapsulation of pollutants (immobilization by fixation), and reduction of solubility and mobility of the toxic substances (immobilization by isolation). Mechanical stabiliza-

* Corresponding author. Tel.: +91 3222 281920; fax: +91 3222 255303.
E-mail address: anjalipal@civil.iitkgp.ernet.in (A. Pal).

tion reduces the bulk mobility of the waste and makes it durable and dimensionally stable. The final product should be capable of resisting deterioration caused by mechanical or environmental stresses.

The extent of mechanical stabilization is evaluated by conducting strength and durability tests. Immobilization by fixation minimizes the local mobility of the individual contaminants. Leaching tests are conducted to determine the effectiveness of the fixation. Immobilization by isolation limits the risk of the contaminants reaching the boundaries of the deposited mass by providing protection against internal contaminants transport and by restricting internal transport pathways (low permeability). Other objectives of solidification and stabilization processes are, of course, the production of a monolithic solid mass and the improvement of the handling and physical characteristics of the waste [3]. Cement-based solidification/stabilization technology currently provides the most promising solution for the disposal of arsenic wastes [5]. Solidification/stabilization with Portland cement is often suggested as the best treatment for wastes that cannot be reduced or recycled [6,7]. In a good solidified product, hazardous contaminants are chemically immobilized by the alkaline environment and in the hydration products the contaminants are physically trapped by encapsulation in the overall bulk of the matrix [8].

In the literature, it is reported that the arsenic waste can be solidified/stabilized with cement, fly ash, and $\text{Ca}(\text{OH})_2$ [7–9]. This is due to the formation of calcite, which seals the pores of the solidified sample and precipitate formation of calcium arsenite occurs where cement acts as a binder [7]. It is also reported that arsenic can be chemically fixed into cementations environment of the solidified/stabilized matrices by three important immobilization mechanisms, viz., sorption onto C–S–H (calcium–silica–hydrate) surface, replacing SO_4^{2-} of ettringite, and reaction with cement components to form calcium–arsenic compounds [10]. It is observed that the early hydration of cement is inhibited by the presence of AsO_4^{3-} , and that the inhibition is mainly caused by the formation of highly insoluble $\text{Ca}_3(\text{AsO}_4)_2$ on the surface of hydrating cement particles [11]. This hydration by-product not only has a direct influence on strength of the solidified matrices but also potentially has a hand in waste immobilization [10]. Solidification of arsenic-laden or other heavy metal-laden solid waste with cement is a suitable technique to reduce leachability of those metals [5–7].

The present study was aimed, for the first time, at evaluating the potentiality of the CalSiCo-sludge to be used as PCC without affecting the environment through leaching of arsenic from PCC under various natural and synthetic conditions.

2. Materials and methods

2.1. Materials

The material CalSiCo for arsenic adsorption was prepared [1] in two stages. In the first stage CaCl_2 , calcium silicate ($x\text{CaO}\cdot\text{SiO}_2$) and distilled water were mixed at a selected proportion and slurry was made. After that the paste was kept in a

Table 1
Chemical compositions (%) of CalSiCo (EDAX-test)

	Compositions	
	1st stage	2nd stage
Al	4.02	5.88
Si	18.65	17.5
Na	1.51	–
S	4.25	7.21
K	1.44	0.43
Ca	65.85	42.47
Mg	0.87	1.27
Cu	0.75	25.24
Fe	0.87	–

mould for 24 h for setting. Then, the hardened paste was separated from the mould and kept in an autoclave for 1 h for steam curing. Subsequently, it was granulated to get particles having 0.251 mm effective size through a set of sieves. The granulated materials were then washed with distilled water and dried in the sun. In the second stage, these granulated materials were soaked in 0.5N CuSO_4 for 2 h in a mechanical stirrer and then these were washed with distilled water and dried in the sun. The elemental compositions of CalSiCo were determined by EDAX (Oxford ISIS, Model S-440) test using scanning speed of $2^\circ/\text{min}$ with C-filtered, $\text{Cu K}\alpha$ radiation at 35 kV and 20 mA. The compositions are summarized in Table 1. Adsorbent particles having an average diameter of 0.251 mm was used throughout the study.

2.2. Preparation of arsenic-laden CalSiCo-sludge

Arsenic-laden CalSiCo-sludge was prepared separately on As(III) and As(V) adsorption. The adsorption was done at a CalSiCo dose of 10 g/l for As(III) and 5 g/l for As(V). Five grams of CalSiCo was mixed with 500 ml of As(III) (concentration 0.4 mg/l) at pH 10.2, and stirred in a mechanical shaker for 6 h. In case of As(V), 2.5 g of CalSiCo was mixed with 500 ml of As(V) (concentration 0.4 mg/l) at pH 7.2, and was shaken for 6 h. The adsorbent doses were selected based on the batch studies. The reported q_e (amount of adsorbate adsorbed per unit weight of adsorbent at equilibrium) obtained from isotherm studies, for As(III) and As(V) at temperature $20 \pm 2^\circ\text{C}$ were 0.352 and 0.876 mg/g, respectively [1].

2.3. Extraction test performed on arsenic-laden CalSiCo-sludge

An appropriate way to examine the effectiveness of the immobilization of the contaminants is to perform extraction tests [3]. As(III) and As(V) containing CalSiCo-sludge were subjected to extraction test under various conditions of leaching.

The sludge material is added to a leaching medium at a chosen ratio of weight of waste material to volume of leachant and shaken for a certain period of time. At the end of this period, equilibrium is reached between the leachant and the sludge material. These tests are generally used to determine the maximum

Table 2
Compressive strength of M15 concrete blocks

Types of block	28-Day compressive strength				MPa
	Trial-1 (<i>t</i>)	Trial-2 (<i>t</i>)	Trial-3 (<i>t</i>)	Average strength (<i>t</i>)	
Original	41.5	41.0	41.5	41.5	18
S-I	39.0	39.0	39.0	39.0	17
S-II	36.0	35.5	36.0	36.0	15.7
S-III	34.5	35.0	35.0	35.0	15.3
S-IV	17.5	17.5	17.5	17.5	7.6

leachable concentration under the chosen conditions of leaching medium and leaching time. In this study, the extraction procedure used was DIN test [3,9]. To test effective leaching at various conditions using DIN test, the following procedure was followed: 11 of distilled water was added to 100 g of CalSiCo-sludge at different pH conditions and in presence of different ions, and then it was shaken for 20–30 days in a closed vessel. To test the effect of various ions on the leachability of arsenic from CalSiCo-sludge it was subjected to distilled water containing NO_3^- , Cl^- , SO_4^{2-} at a constant pH (in the range of 6–8). To mimic the natural systems the leachability of arsenic from the CalSiCo-sludge by tap water and rain water were also tested. During the test, the pH of the leachate was checked and maintained throughout the leaching period.

2.4. Preparation of concrete

M15 (1:2:4) (M15 means the design strength of the concrete is 15 MPa, and the ratio in the parentheses indicates the ratio of cement, sand, and coarse aggregates). Concrete cube of 150 mm side was prepared replacing 10, 25, 35 and 50% of the total cement required with exhausted CalSiCo-sludge and they were designated as S-I, S-II, S-III and S-IV, respectively. The water to cement (W/C) ratio was 0.48. This particular ratio was maintained, as per the Bureau of Indian Standards (BIS) 456:2000 [12], to obtain optimum strength as well as better workability of the concrete. Three sets of concrete blocks were prepared for each of S-I–IV, and for the original (without CalSiCo-sludge mixed) M15 concrete block. All blocks were kept in different water container for 28 days curing, and the tests were done during this period. The comparative compressive strength of the five concrete blocks has been given in Table 2.

2.5. Extraction test performed on PCC containing arsenic-laden CalSiCo-sludge

To test effective leaching at various conditions using DIN test, the following procedure was followed: about 11 of distilled water was added to 100 g of granulated (effective size of 2.5 mm) concrete blocks under strongly acidic conditions (in the pH range of 1–3) and under strongly alkaline conditions (in the pH range of 13–14), and also in presence of Cl^- ions at different concentration levels. After that it was shaken for 20–30 days in a closed vessel.

2.6. Analytical methods

The concentration of As(III) and As(V) in leachate was measured by standard silver diethyldithiocarbamate (SDDC) method [13]. For each test 35 ml of sample was taken in a clean Gutzeit generator and to that 5.0 ml concentrated HCl, 2.0 ml KI solution (15%, w/v) and 0.4 ml of SnCl_2 (40%, w/v) were added successively. Then, 15 min time was allowed for the reduction of As(V) to As(III). In the mean time the glass wool in the scrubber was impregnated with six to eight drops of lead acetate solution and 4.0 ml SDDC reagent was taken in the absorber tube. After the 30 min reaction, 3.0 g zinc was added into the generator and immediately scrubber–absorber assembly was connected. The reaction was allowed to continue for another 30 min to ensure complete evolution of AsH_3 gas. It is also prescribed to heat the generator slightly to ensure that all arsine had been released. The absorbance of the SDDC reagent was measured at 535 nm using the reagent blank as the reference.

3. Results and discussion

3.1. Leaching of arsenic from CalSiCo-sludge

Leaching of As(III) and As(V), under various pH conditions, from CalSiCo-sludge prepared on separate adsorption of As(III) and As(V), were shown in Figs. 1 and 2, respectively. It is observed that the leaching of As(III) is above 0.05 mg/l at any pH. But in case of As(V) the leaching is above 0.05 mg/l only at

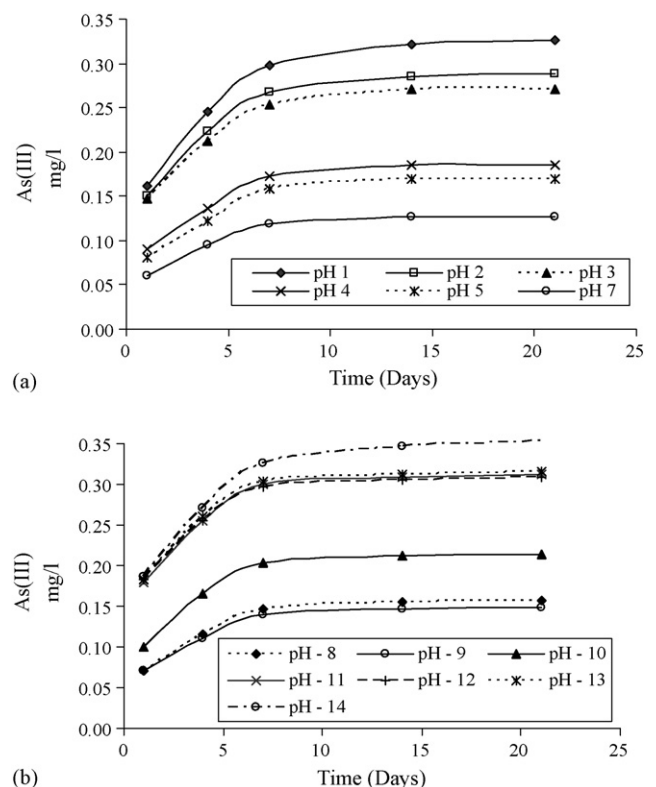


Fig. 1. Leaching of As(III) from CalSiCo-sludge at different pH: (a) pH 1–7 and (b) pH 8–14.

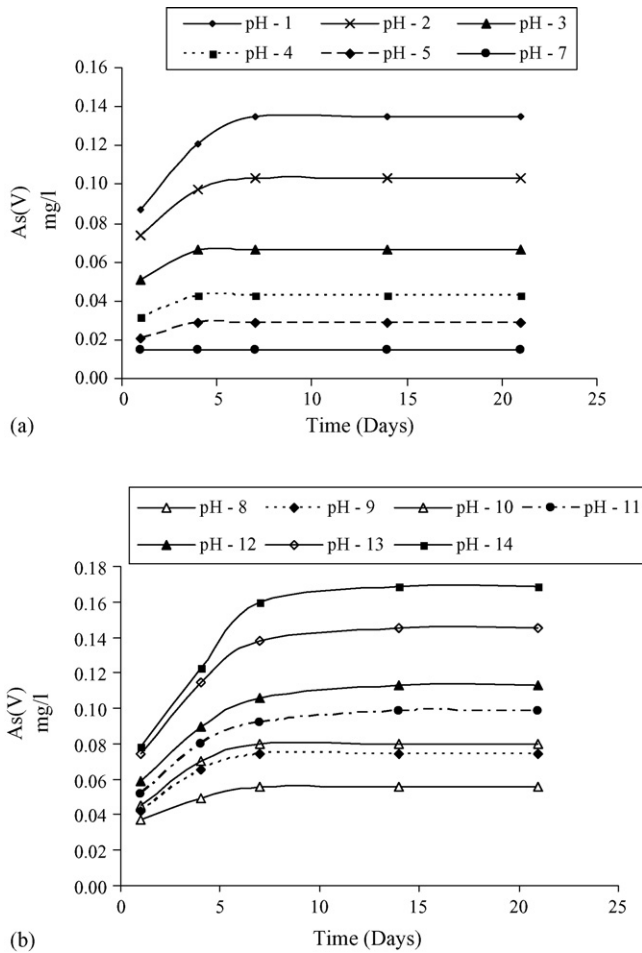


Fig. 2. Leaching of As(V) from CalSiCo-sludge at different pH: (a) pH 1–7 and (b) pH 8–14.

pH > 8 and at pH < 4. Within the range of pH 4–8, however, the leaching of As(V) is in the range of 0.02–0.05 mg/l. The pH_{ZPC} of CalSiCo (i.e. the pH at which it bears the zero charge) is 7.6. During the preparation of CalSiCo-sludge, As(III) is adsorbed on CalSiCo at pH ~ 10. At this pH As(III) stays mostly as arsenite oxyanion. In case of As(V) almost in the entire range of pH, it stays in its ionic form. The adsorption of As(V) is carried out at pH ~ 7, which is very close to the pH_{ZPC} of CalSiCo (i.e. 7.6), and at this pH the surface of the adsorbent almost carries no charge.

In order to understand the sorption type, the equilibrium data was fitted into the linearized Dubinin–Radushkevich (D–R) isotherm, which has been expressed by Eq. (1) [14]:

$$\ln Q = \ln Q_m - k\varepsilon^2 \quad (1)$$

where ε (Polanyi potential) = $RT \ln(1 + 1/C_e)$, Q the amount of arsenic adsorbed at equilibrium per unit weight of CalSiCo (mg/g), Q_m the theoretical saturation capacity, C_e the equilibrium concentration of arsenic in solution (mg/l), k a constant related to the adsorption energy ($\text{mol}^2 \text{kJ}^{-2}$), R the gas constant ($\text{kJ mol}^{-1} \text{K}^{-1}$) and T is the temperature (K).

The values of the D–R isotherm constants k and Q_m were calculated from the slope and intercepts of the linear plot of $\ln Q$

against ε^2 . From Eq. (1), the calculated k values for As(III) and As(V) were 0.0751 and $0.0317 \text{ mol}^2 \text{kJ}^{-2}$, respectively.

The mean free energy of adsorption (E), defined as the free energy change when one mole of ion is transferred from infinity in solution to the surface of the solid, was calculated from the k value using Eq. (2):

$$E = -(2k)^{-0.5} \quad (2)$$

The calculated values of E are found to be 2.58 kJ mol^{-1} for As(III) and 3.97 kJ mol^{-1} for As(V). The magnitude of E gives information about the type of adsorption and if this value lies between 8 and 16 kJ mol^{-1} , the adsorption type is ion-exchange. But in the present case, the values of E for both As(III) and As(V) lie within the energy range of physical adsorption, which is due to weak Van der Waals force.

The leaching of arsenic is more in case of As(III)-laden CalSiCo-sludge compared to that of As(V)-laden CalSiCo-sludge at any pH value, possibly because, physisorption is more predominant in case of As(III) adsorption. In case of As(V), it leaches out at low pH (<4), because at this pH As(V) exists mostly as the non-ionic form. At higher pH, however, OH^- competes in the adsorption process and adsorbed arsenate anion leaches out from the adsorbent.

The leaching test in presence of Cl^- ions showed (Fig. 3) that maximum leaching occurs when extraction liquid contains Cl^- and leaching of arsenic increases with the increase in the Cl^- ion concentration. Leaching of arsenic from CalSiCo-sludge in presence of NO_3^- and SO_4^{2-} has been shown in Figs. 4 and 5,

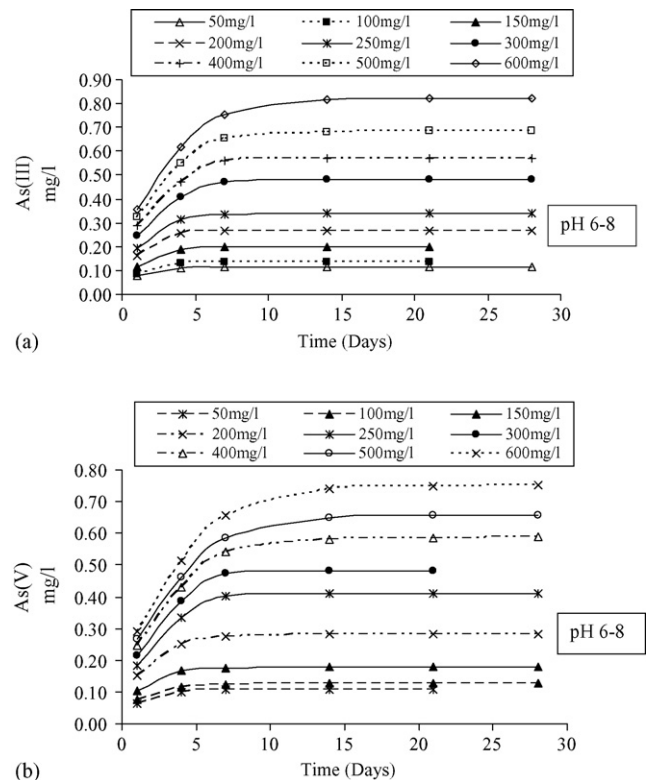


Fig. 3. Leaching of (a) As(III) and (b) As(V) from CalSiCo-sludge at different Cl^- concentrations.

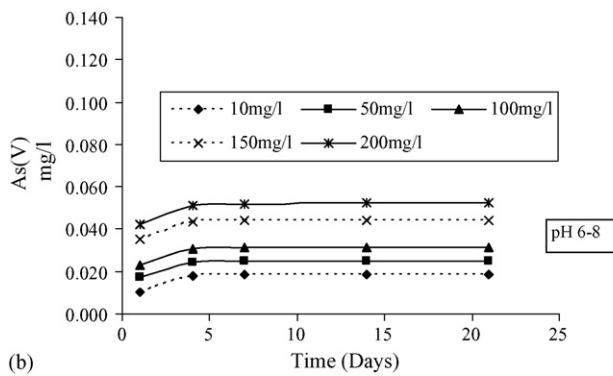
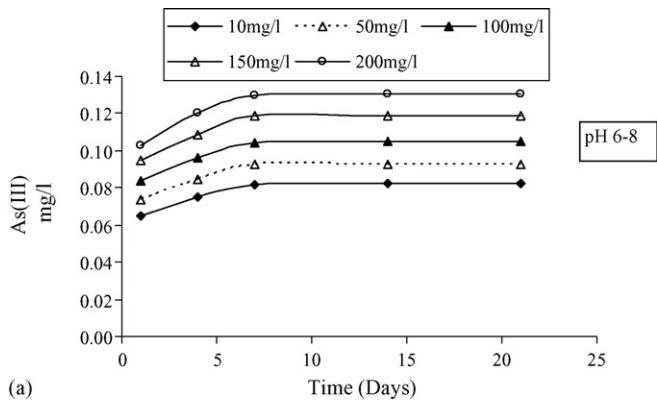


Fig. 4. Leaching of (a) As(III) and (b) As(V) from CalSiCo-sludge at different NO₃⁻ concentrations.

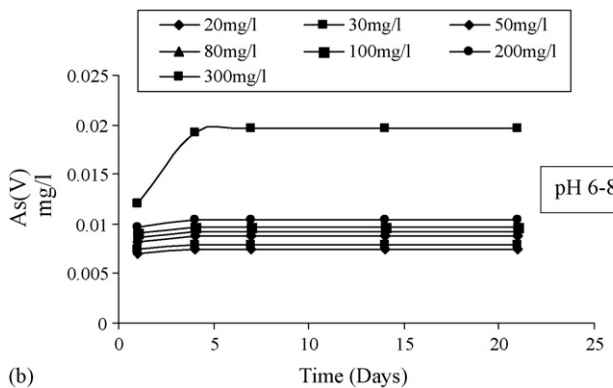
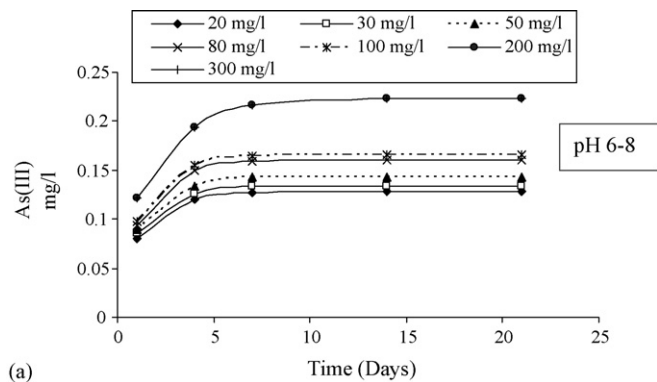


Fig. 5. Leaching of (a) As(III) and (b) As(V) from CalSiCo-sludge at different SO₄²⁻ concentrations.

Table 3
Characteristics of tap water and rain water

Parameters	Tap water	Rain water
pH	6.8–7.3	5.0–6.5
Turbidity	0–5 NTU	<10 NTU
Conductivity	(2.4–2.48) × 10 ⁻³ mho/cm	(3.1–3.5) × 10 ⁻³ mho/cm
Hardness	<100 mg/l as CaCO ₃	<50 mg/l as CaCO ₃
TDS	<100 mg/l	<200 mg/l
Fe ²⁺	<0.1 mg/l	–
Chloride	<30 mg/l	20–70 mg/l

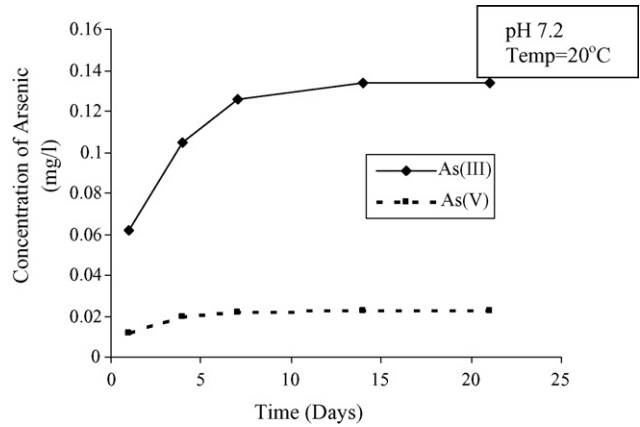


Fig. 6. Leaching of As(III) and As(V) from CalSiCo-sludge in tap water.

respectively. It is observed that leaching of arsenic in these cases is almost equal to the leaching of arsenic at pH of 6–8. So it can be concluded that NO₃⁻ and SO₄²⁻ have no significant effect on the leaching of arsenic from the CalSiCo-sludge.

To observe the leaching of arsenic in natural systems, studies have been carried out in both tap water and rain water. The characteristics of tap water and rain water have been given in Table 3. The results are shown in Figs. 6 and 7, respectively. It is observed from the figures that tap water has no effect on leaching of arsenic while rain water causes significant amount of leaching of arsenic from exhausted CalSiCo-sludge. The strong effect of rain water in leaching is due to the pH of rain water (pH 5.5) and the results are comparable to the leaching of arsenic in distilled water at pH ~ 5.0 condition. However, the leaching of As(V) is much less compared to the leaching of As(III).

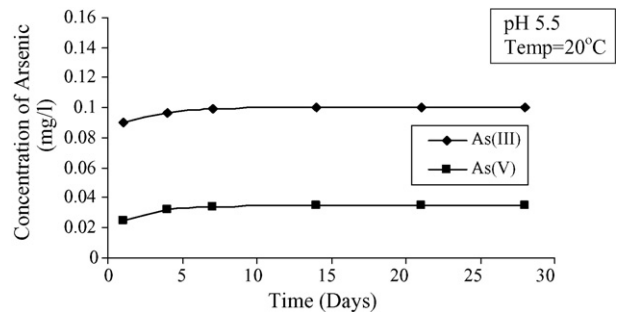


Fig. 7. Leaching of As(III) and As(V) from CalSiCo-sludge in rain water.

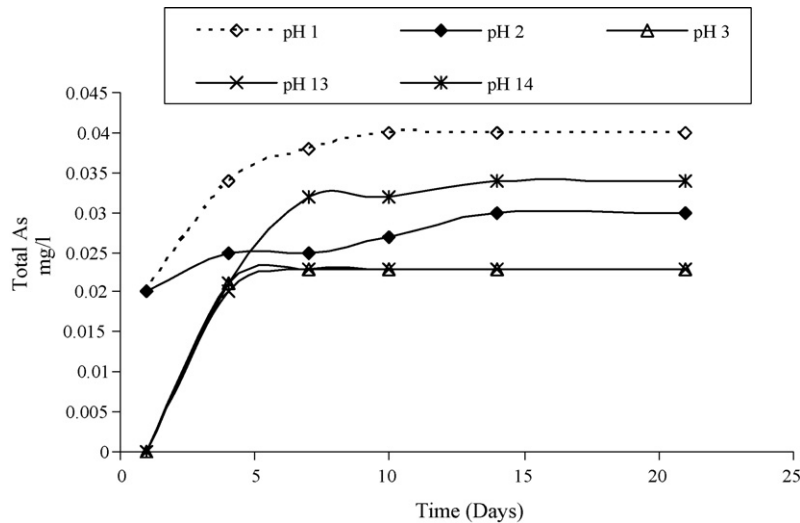


Fig. 8. Leaching of arsenic from S-III at different pH conditions.

3.2. Leaching of arsenic from PCC

The main objective of our studies was to see whether the leaching could be minimized in PCC, so that the sludge can be used for the construction purpose. Thus, leaching test was conducted on PCC under different pH conditions and in presence of Cl⁻ ions at different doses. No leaching of arsenic (total) was observed from S-I–IV even if the leaching medium contains high dose of Cl⁻ (600 mg/l). It is possibly due to the formation of CaHAsO₄, CaHAsO₃ and Ca₃(AsO₄)₂ [9–11] by the reaction of arsenic and the hydrated products (Ca(OH)₂, calcium–silica–hydrate gel) of Portland cement. It is also due to the encapsulation of arsenic within the micro pores of the amorphous gel structure of calcium–silica–hydrate (C–S–H) [10]. No leaching of arsenic was observed from S-I under the entire range of pH. Only arsenic leaching at a concentration level of 0.02 mg/l was observed from S-II under strongly acidic condition (pH ~ 1). It was also observed that leaching of arsenic is zero from S-I–IV even at highly alkaline condition (pH ~ 12). Though the leaching of arsenic is observed from S-III and S-IV under various pH conditions (Figs. 8 and 9) but it is very less

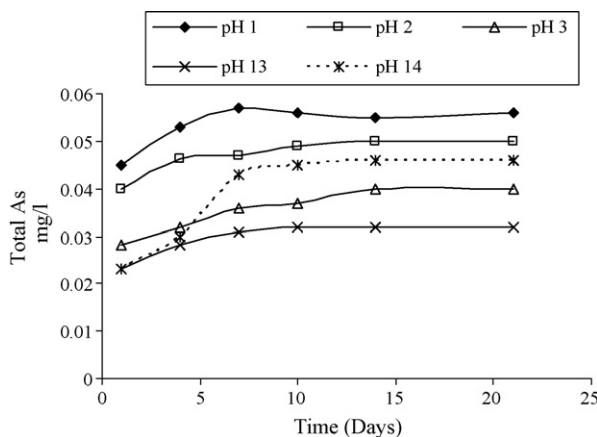


Fig. 9. Leaching of arsenic from S-IV at different pH conditions.

in comparison to that of CalSiCo-sludge. The slight leaching is due to the corrosion and weakening effect of acid on physical cement matrix [6].

3.3. Mathematical formulation for the determination of rate of leaching from the M15 grade concrete containing CalSiCo-sludge

Leaching of contaminants into the leaching water results from the mass transport of the contaminants contained in the solidified waste. The following different transport processes such as diffusion, dissolution, ion exchange, corrosion and surface effects may occur during the leaching of arsenic from the concrete block containing CalSiCo-sludge. It is believed that leaching of contaminants out of the cement-based waste is mostly a diffusion-controlled process [3]. The solution of the diffusion equation depends on the initial and boundary conditions. The cement-based waste is assumed to be a semi-infinite medium, which implies that the mass of the constituents removed from the waste is negligible in comparison to the total mass in the waste [less than 20% of a leachable species is allowed to leach under these assumptions (Eqs. (3) and (4))]. The contaminants are initially assumed to be uniformly distributed in the solidified waste and to have a zero surface concentration in the surrounding medium once leaching begins.

With the assumption of a constant diffusion co-efficient, the diffusion flux (*J*) across the solid/solution interface can be expressed [5] as

$$J = \frac{dMt}{dS dt} = \sqrt{\frac{D_e}{\pi t}} C_0, \tag{3}$$

with *C*₀ is the initial concentration of the leaching substance in the waste specimen (mg/cm³), *D*_e the effective diffusion co-efficient (cm²/s) and *t* is the leach time (s).

The total amount of leaching substance that has diffused out of the medium surface at time *t*, *Mt*, can be derived from Eq. (3)

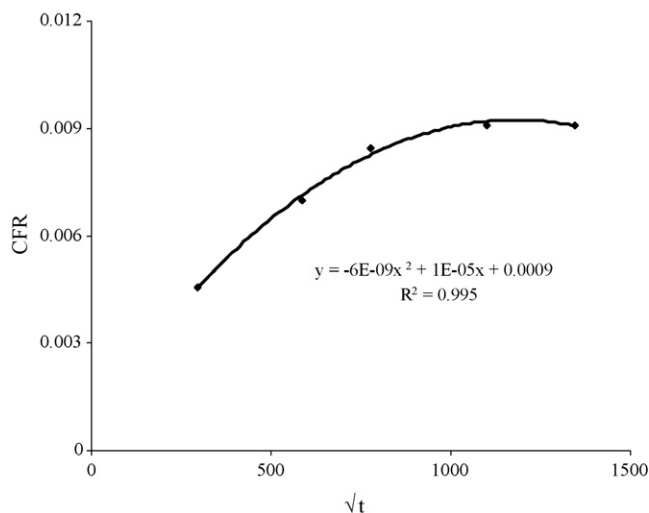


Fig. 10. Plot of \sqrt{t} vs. CFR for S-IV at pH 1.

by integration over time and surface area as follows:

$$\int dMt = \int_0^t \int_0^S \left[\sqrt{\frac{D_e}{\pi t}} \right] C_0 dS dt \quad \text{or} \quad Mt = 2M \frac{S}{V} \sqrt{\frac{D_e t}{\pi}} \quad (4)$$

where M is the total initial amount of leaching substance in the waste specimen (mg), S the surface area of the specimen (cm^2) and V is the volume of the specimen (cm^3).

The cumulative fraction of leaching substance that has been released from solidified waste at time t (CFR) is defined as the ratio of Mt to M and is given by

$$\text{CFR} = \frac{Mt}{M} = \frac{2}{\sqrt{\pi}} \frac{S}{V} \sqrt{D_e t} \quad (5)$$

This Eq. (5) is linear with square root of time, and the slope of this plot (\sqrt{t} versus CFR) relates to the effective diffusion co-efficient. A plot of \sqrt{t} versus CFR for S-IV at pH 1 is shown in Fig. 10. D_e values generally range from $10^{-5} \text{ cm}^2/\text{s}$ (very mobile) to $10^{-15} \text{ cm}^2/\text{s}$ (immobile), but in our case from the figure the value of D_e has been found out as $8.75 \times 10^{-16} \text{ cm}^2/\text{s}$. The very low value of D_e in the order of 10^{-16} indicates that the arsenic is highly immobilized after incorporating arsenic-laden CalSiCo-sludge within the matrix of cement concrete. It is, however important to mention in this context that although with 50% cement replacement a very low D_e value is obtained but from the aspect of applicability of concrete it is not suitable as the strength of concrete goes 50% of the target strength (Table 2). Maximum of 35% replacement is encouraged considering strength as well as leaching of arsenic. It can be concluded from the results obtained (Table 4) that at the beginning of the leaching diffusion is not only the governing process but also other processes like velocity gradient, dissolution, ion exchange, corrosion and surface effects may be taking place. As the leaching proceeds other processes gradually stop and diffusion takes upper hand.

Table 4
Comparison of Mt values for S-IV at pH 1

Day	Experimental value (mg)	Theoretical value from Eq. (5) (mg)	% of variation from experimental value
1	0.0080	0.0043	-46.25
4	0.0123	0.0083	-32.5
7	0.0149	0.011	-26.17
14	0.0160	0.015	-6.25
21	0.0160	0.018	+12.5

4. Conclusion

Leaching of arsenic from arsenic-laden CalSiCo-sludge under various conditions has been tested. Management of arsenic-laden CalSiCo-sludge is of utmost importance as in most of the cases the leaching of arsenic was more than the maximum permissible limit of arsenic in drinking water. Leaching of As(III) is found to be more than that of As(V) in all cases. Higher leaching of arsenic from CalSiCo-sludge takes place when pH of the surrounding medium reaches <3 , or >11 , and also in presence of Cl^- ions. Preparation of cement concrete seems to be a viable option for CalSiCo-sludge management. On stabilization/solidification, calcium ions in the Portland cement form different complexes that are insoluble in water. It is important to maintain the ratio of cement to CalSiCo-sludge to achieve the desired strength of cement concrete and to stop the arsenic leaching under various conditions. In the present study solidification/stabilization of arsenic-laden CalSiCo-sludge is observed to make arsenic highly immobilized after treating with cement concrete. Maximum of 35% cement replacement with arsenic-laden CalSiCo-sludge can be done without compromising strength of the concrete.

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