

Examination of solidified and stabilized matrices as a result of solidification and stabilization process of arsenic-containing sludge with portland cement and lime

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Abstract

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By solidification and stabilization (S/S) with Portland cement and lime, it is possible to reduce arsenic concentration in leachate of the arsenic-containing sludge from arsenic removal process by coagulation with ferric chloride. From the initial arsenic concentration in leachate of unsolidified /unstabilized sludge which was around 20.75 mg/L, the arsenic concentrations in leachate of solidified/stabilized waste were reduced to 0.3, 0.58, 1.09, and 1.85 mg/L for the waste-to-binder ratios of 0.15, 0.25, 0.5, and 1, respectively, due to the formation of insoluble calcium-arsenic compounds. To be more cost effective for the future, alternative uses of these S/S products were also assessed by measurement of compressive strength of the mortar specimens. It was found that the compressive strengths of these matrices were from 28 ksc to 461 ksc. In conclusion, considering compressive strength and leachability of the solidified matrices, some of these solidified/stabilized products have potential to serve as an interlocking concrete paving block.

Key words : solidification/stabilization, arsenic-iron hydroxide sludge, waste utilization, leaching test, scanning electron microscopy, FT-IR

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บทคัดย่อ

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การศึกษากรองหน่อแข็งจากกระบวนการหน่อแข็งและปรับเสถียรกากตะกอน
ที่ปนเปื้อนสารหนูด้วยปูนซีเมนต์และปูนขาว
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จากความเข้มข้นเริ่มต้นของสารหนูในน้ำชะละลายของกากตะกอนปนเปื้อนสารหนูที่ไม่ได้ปรับเสถียรซึ่งเกิดจากกระบวนการผลิตน้ำดื่มโดยการโคแอกกูเลชันด้วยเฟอร์ริกคลอไรด์ ที่มีค่าเท่ากับ 20.75 มก/ลิตร กระบวนการหน่อแข็ง และ ปรับเสถียรโดยใช้ปูนซีเมนต์ และ ปูนขาวเป็นวัสดุประสาน สามารถลดความเข้มข้นของสารหนูในน้ำชะละลายของกากตะกอน ไปได้ถึง 0.3, 0.58, 1.09, และ 1.85 ที่อัตราส่วน ตะกอน ต่อ วัสดุประสาน เท่ากับ 0.15, 0.25, 0.5, และ 1 ตามลำดับ เนื่องจากการก่อตัวของ สารประกอบแคลเซียม และ สารหนูที่ไม่ละลายน้ำ นอกจากนี้ความเป็นไปได้ในการใช้ประโยชน์จากหน่อแข็งเหล่านี้ยังถูกประเมินโดยวัดกำลังรับแรงอัดของก้อนตัวอย่างที่ 28 วัน ซึ่งพบว่ากำลังรับแรงอัดมีค่าตั้งแต่ 28 ถึง 461 กก/ตร.ซม ซึ่งเมื่อพิจารณาจากทั้งความเข้มข้นของสารหนูในน้ำชะละลาย และ ค่ากำลังรับแรงอัด พบว่า ก้อนหน่อแข็ง และ ปรับเสถียร บ้างก่อนสามารถนำไปใช้ประโยชน์เป็นก้อนคอนกรีตประสานปูนพื้นได้

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Named "the largest mass poisoning of a population in history" (Khundkar, 2003), the contamination of groundwater by arsenic in Bangladesh has been a point of attention and become a global issue. In reality, not only Bangladesh but also many countries throughout the world such as Argentina, Chile, China, Hungary, India (West Bengal), Mexico, Pakistan, Thailand, USA, and Viet Nam have been facing the same problem (Adeel and Ali, 2003).

True to its nickname, 'the silent killer' (Adeel and Ali, 2003), arsenic hides behind the consumable appearance of groundwater in contaminated shallow aquifers. Due to the fact that arsenic dissolved in water has no color, smell, or taste, it is not surprising that the poor villagers in the affected areas have drunk this poison for decades without awareness of being at risk of diseases ranging from melanosis to skin cancer and gangrene (Harunur-Rashid and Abdul Karim Mridha, 1998).

In order to decrease the severity of this problem, several arsenic removal technologies have

been applied in those regions. Each technology has its own advantages and disadvantages. However, one of the economical technologies (Meng *et al.*, 2001) usually used in Asian countries is coagulation with ferric chloride because it is suitable for poor villagers' situation in this region.

Consequently, arsenic in water transfers to amorphous ferric hydroxide, the main product of the coagulation process, and therefore the by-product of this arsenic removal process is arsenic-containing sludge. The amount of arsenic in the sludge may vary widely. It was estimated that the range of total arsenic in the sludge was from 700 to 15,000 mg/kg, depending on initial arsenic concentration, finished water concentration, and background coagulant demand (Frey *et al.*, 2003). Therefore, there is the potential for some of the sludge to present a threat to the environment and public health if improperly managed. For example, if the hazardous sludge is dumped together with municipal solid waste in sanitary landfills, large amounts of arsenic in the sludge may leach out

and ultimately contaminate the groundwater.

For this reason, in this study, the solidification/stabilization (s/s) of synthesized arsenic iron hydroxide sludge, which simulated the sludge produced from arsenic removal by the coagulation process with ferric chloride, by the combination of two binding agents, Portland cement and lime, was investigated. The potential to utilize the solidified/stabilized products was evaluated under the criteria of their compressive strength requirement and regulatory leachate concentration limit. Moreover, Scanning Electron Microscopy (SEM) together with Energy Dispersive Spectroscopy (EDS) was used to gain microstructure information for better understanding about how arsenic-containing sludge affected microstructures of cement hydration products as well as how it influenced the unconfined compressive strength of the solidified/stabilized matrix. In addition, Fourier Transform Infrared Spectroscopy (FT-IR) was also used to confirm the negative effect of arsenic-iron hydroxide sludge on cement hydration by tracking the development of the polymerization of the orthosilicates.

Materials and Methods

1. Solidification/stabilization

The hazardous, synthesized arsenic-iron hydroxide sludge, of which arsenic concentration in leachate according to the leaching procedure under the Notification of the Ministry of Industry No. 6, B.E. 2540, LP-No.6, (DIW, 1997) was 20.75 mg/L, was used in this study. The leachate concentration of this untreated sludge is 5 times higher than the acceptable limit which is 5 mg/L. Portland Cement Type I (Elephant Brand®) and hydrated lime, calcium hydroxide (analytical reagent grade, UNIVAR), were used as binding agents. To lessen leachability and to improve physical property of the hazardous arsenic-containing sludge, ten solidification/stabilization recipes were investigated. The recipes differed from one another in waste-to-binder, Portland cement-to-lime, and water-to-binder ratios. For each S/S recipe, three replicates

were cast with the weight of cement to sand ratio of 1.00:2.75 according to ASTM C109-95, for 2x2x2-inch mortar. All 30 S/S matrices were tested for their unconfined compressive strengths (UCS) at curing age of 28 days in order to examine each recipe's potential for utilization as construction material. The mixture proportions of mortar are shown in Table 1.

2. Scanning Electron Microscopy equipped with Energy X-ray Spectroscopy (SEM-EDS)

Ten samples with the same mixture proportion as those of Table 1 but without sand were cast into 5 mm-diameter plastic tubes. After 28 days, the samples were cross-sectionally cracked and coated with gold. Then, their morphology was observed by JSM 6400 Scanning Electron Microscopy. Moreover, EDS, Link^{isis} Series, was used to identify various microstructures in these solidified waste forms by determining their elemental compositions.

3. Fourier Transform Infrared Spectroscopy (FT-IR)

The same set of cement paste, analyzed by SEM-EDS, was subjected to FT-IR. All FT-IR spectra in the present study were recorded by BIO-RAD Fourier Transform Infrared Spectroscopy using potassium bromide pellets at sample-to-KBr ratio of 1:50. The spectra were recorded under the wavenumber of 400-4000 cm⁻¹ with 8 cm⁻¹ resolution and 200 scans each time.

4. Leaching procedures

In this present study, all solidified/stabilized products were crushed to a particle size smaller than 9.5 mm and subjected to the leaching procedure under the Notification of the Ministry of Industry No. 6, B.E. 2540, LP-No.6, (DIW, 1997). Then, the leachate was analyzed by Inductive Coupled Plasma-Optical Emission Spectroscopy (ICP-OES), a multi-element analytical technique of which typical 3 sigma detection limit is 3 µg/L for arsenic at the wavelength of 188.98 nm.

Table 1. Mix proportions of mortar specimens

Notation*	Mixture proportion (kg/m ³)					Ratio		
	Water (w)	Cement (c)	Lime (l)	SSD Sludge ⁺ (s)	SSD Sand	w/(l+c) [#]	s/(l+c)	l/(l+c)
CW4-S00-L00	217.53	543.99	0.00	0.00	1495.82	0.40	0.00	0.00
SW4-S15-L00	210.65	526.69	0.00	78.97	1448.25	0.40	0.15	0.00
SW4-S25-L00	206.32	515.80	0.00	128.95	1418.29	0.40	0.25	0.00
SW4-S50-L00	196.07	490.33	0.00	245.09	1348.29	0.40	0.50	0.00
SW4-S100-L00	177.97	446.28	0.00	446.28	1227.35	0.40	1.00	0.00
CW9-S00-L04	376.60	251.01	167.39	0.00	1150.46	0.90	0.00	0.40
SW9-S15-L04	367.31	244.76	163.23	61.19	1122.11	0.90	0.15	0.40
SW9-S25-L04	361.38	240.92	160.67	100.44	1104.01	0.90	0.25	0.40
SW9-S50-L04	347.28	231.47	154.42	193.02	1061.24	0.90	0.50	0.40
SW9-S100-L04	322.30	214.81	143.21	358.18	984.83	0.90	1.00	0.40

* The notation of each S/S recipe consists of three sections e.g. CW4-S00-L00. The first alphabet of the first section is used to identify type of S/S recipes, between the control recipes that start with C and the S/S recipes with the addition of the sludge that start with S. The second and the third alphabets of the first section are used to indicate water-to-binder ratio of each recipe. For example, W9 means that the water-to-binder ratio is equal to 0.9 while W4 means that the water-to-binder ratio is equal to 0.4. The second section of the notation is used to indicate SSD sludge-to-binder ratio. For example, S15 means that SSD sludge-to-binder ratio is equal to 0.15 while S50 means that SSD sludge-to-binder ratio is equal to 0.5, and so on. The last section of the notation is used to indicate lime-to-binder ratio. For example, L00 means that lime-to-binder ratio is equal to zero, or no lime was added into this S/S recipe. In contrast, L04 means that lime-to-binder ratio is equal to 0.4.

+ SSD sludge was composed of dried sludge (45.66 % by weight) and sorbed water (54.34% by weight).

Binder consists of lime and cement; therefore, total amount of binder in each S/S recipe is the summation of amount of lime and cement.

Table 2. USC (ksc) of each recipe without lime

Notation	Average UCS (ksc)
CW4-S00-L00	461.06
SW4-S15-L00	428.81
SW4-S25-L00	371.82
SW4-S50-L00	269.77
SW4-S100-L00	144.84

Table 3. USC (ksc) of each recipe with lime

Notation	Average UCS (ksc)
CW9-S00-L04	95.06
SW9-S15-L04	73.02
SW9-S25-L04	54.67
SW9-S50-L04	43.77
SW9-S100-L04	28.81

Results and Discussions

1. Effect of waste-to-binder ratios on unconfined compressive strength

The results of the unconfined compressive strength (UCS) test of all the solidified/stabilized

waste forms are shown in Tables 2 and 3. In addition, Figure 1 illustrates the relationship between waste-to-binder ratios and the UCS of the S/S matrices. It is clearly seen that, for the recipes both with and without addition of lime, the more substitution with the sludge, the lower compressive

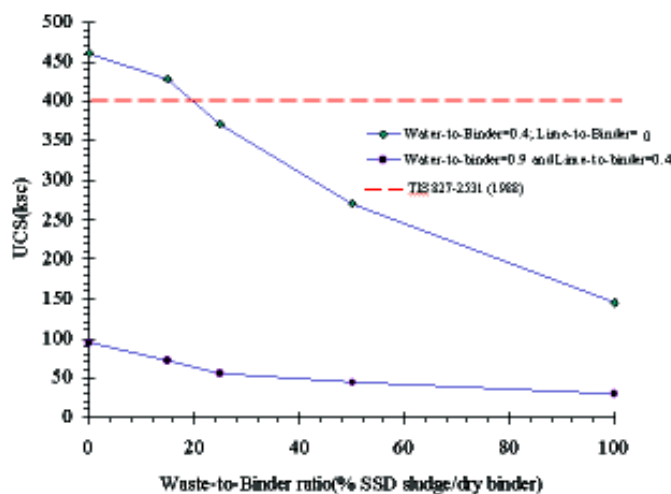


Figure 1. Relationship between Waste-to-Binder Ratio and UCS

strength. For this reason, substitution of Portland cement with the sludge is supposed to cause negative effect to the mechanical property of the solidified products.

According to the available information together with literature, it is supposed that there are two factors contributing to the decline of this mechanical property. Firstly, the substitution with the sludge means the reduction of amount of Portland cement undergoing hydration reaction, which is directly proportional to the UCS. It is therefore not surprising that the higher substitution will result in lower UCS.

Secondly, according to the recent study (Mollah *et al.*, 1998), arsenic can react with calcium to form insoluble calcium-arsenic compounds, which are likely to be able to block hydration development. In the same way, by SEM-EDS, the morphology of a compound believed to be one of the calcium-arsenic compounds forming in a solidified matrix was detected and shown in Figure 2(a). According to its EDS analysis in 2(b), it is found that this calcium arsenic compound is composed of 30.6% O, 26.31% Ca, 20.52% As, 16.26% Fe, 2.83% Si, and 1.80% Al by atomic weight. This microstructure is in good agreement with some of those reported by Bothe and Brown (1999), who investigated the morphologies of

$\text{Ca}_4(\text{OH})_2(\text{AsO}_4)_2 \cdot 4\text{H}_2\text{O}$, $\text{Ca}_3(\text{AsO}_4)_2 \cdot 3^{2/3}\text{H}_2\text{O}$, $\text{Ca}_3(\text{AsO}_4)_2 \cdot 4^{1/4}\text{H}_2\text{O}$, and $\text{Ca}_5(\text{AsO}_4)_3\text{OH}$ by SEM micrographs. They revealed that the morphologies of several of calcium-arsenic compounds except $\text{Ca}_5(\text{AsO}_4)_3\text{OH}$ appeared likely to be plate or leafy crystals. These morphologies have high potential to disturb hydration progress by precipitating onto the surface of unhydrated or partially hydrated cement grains. For example, Figures 2 (c) and (d) depict the morphologies of hydrated cement in the control sample and its EDS spectrum; however, these morphologies were absent from Figures 2 (a) and (b) illustrating the microstructure of the solidified/stabilized waste form. The possible explanation is that the precipitated calcium-arsenic compounds wrapped all the unhydrated or partially hydrated structures.

In addition to the structural characterization technique such as SEM-EDS, the molecular characterization technique such as FT-IR is applicable to examining the calcium-arsenic compound formation potential and the hydration inhibition by considering the polymerization of the orthosilicates in cement.

Mollah *et al.* (1998) noticed the hydration retardation in Portland cement type-V with the presence of sodiumarsenate heptahydrate ($\text{Na}_2\text{HASO}_4 \cdot 7\text{H}_2\text{O}$). They found that the Si-O stretching

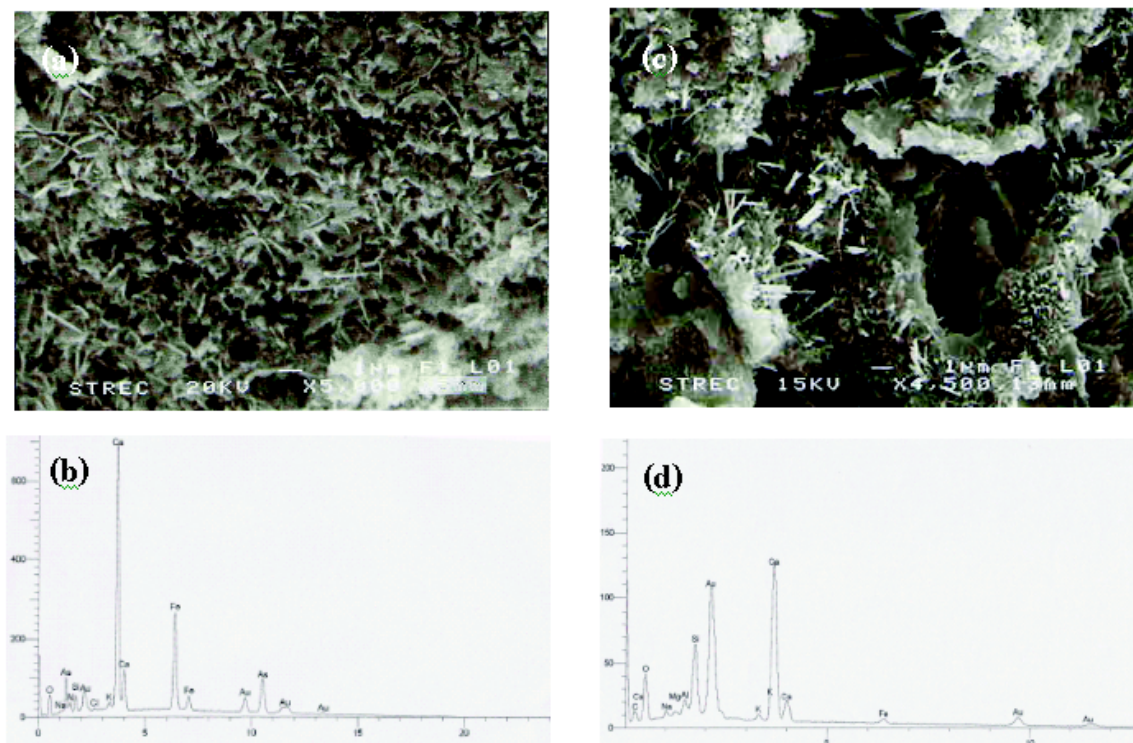


Figure 2. (a) and (b) SEM micrograph and EDS spectra depicting the leafy crystals of insoluble calcium arsenic compound which may act as the insoluble layer covering hydrated and unhydrated cement gains while (c) and (d) SEM micrograph and EDS of hydrated cement without addition of the sludge

band in the As-doped sample appeared at $950\text{--}970\text{ cm}^{-1}$, while that of a control sample without the addition of arsenic salt appeared at $975\text{--}980\text{ cm}^{-1}$. The decline of the Si-O stretching band in the As-doped sample in comparison to that of the control sample suggested that the decline of polymerization of the orthosilicate in cement took place which means that hydration retardation occurred in the As-doped sample.

The same phenomenon mentioned in the previous paragraph was also found in S/S of the arsenic containing sludge in the present study. According to the FT-IR spectrum of unhydrated cement used in this study, the adsorption band at 921 cm^{-1} is due to asymmetric Si-O stretching of alite. This band is in good agreement with that (925 cm^{-1}) reported by Cocke and Mollah (1993).

In contrast to the spectrum of unhydrated cement, Figure 3 illustrates FT-IR spectra of the control sample and the S/S recipes without lime focused from the wavenumber of $800\text{ to }1100\text{ cm}^{-1}$ at the curing age of 28 days. The band at 972 cm^{-1} of the control sample is due to the formation of C-S-H. This band is supposed to shift from the band at 921 cm^{-1} of unhydrated cement due to the polymerization of the orthosilicates in cement by hydration. Therefore, the shifting of Si-O asymmetric stretching band of CW4-S00-L00, the control sample without lime, due to hydration at the curing age of 28 days is 51 cm^{-1} . In contrast, the Si-O asymmetric stretching bands of SW4-S25-L00, SW4-S50-L00, and SW4-S100-L00 are shifted to $964, 968,$ and 966 cm^{-1} , respectively. Therefore, the shifting values due to hydration in the presence of

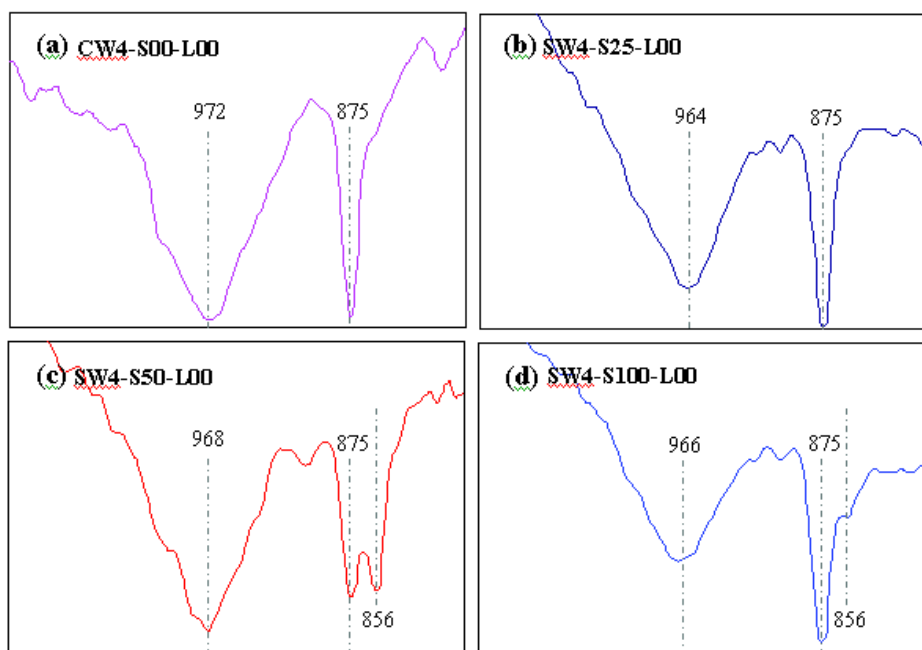


Figure 3. The FT-IR spectrum of the control and S/S recipes without lime focusing on the wavenumber from 800 to 1100 cm^{-1}

the sludge are 43, 47, and 45 cm^{-1} , respectively. Thus, the smaller shifting of Si-O asymmetric stretching bands of the S/S recipes without lime in comparison to that of the control sample indicates that the hydration of cement was retarded in the presence of the arsenic-iron hydroxide sludge. The retardation of the S/S samples corresponds to the additional band at 856 cm^{-1} which is in good agreement with that (860 cm^{-1}) reported by Jing *et al.* (2003). This band represents As(V)-O-Ca bonds (Jing *et al.*, 2003); therefore, it can be a good evidence to confirm the existence of calcium-arsenic compounds.

2. Leachability of the Solidified/Stabilized Products

The results of the leaching test on the eight different S/S recipes as well as two control samples are illustrated in Figures 4 (a), (b), and (c) in which the concentrations (mg/L) of As, Ca, and pH of leachate are shown, respectively.

Considering the available information, it

is found that two obvious issues should be taken into consideration. The first issue arises when the comparison between concentration of arsenic in leachate of the two different kinds of the S/S matrices, with and without addition of lime, is made. As shown in Figure 4 (a), it is found that the concentrations of arsenic in both cases at the same waste-to-binder ratio are not significantly different. Theoretically, waste immobilization involves two broad mechanisms, physical and chemical immobilization. The main physical mechanism focused in the present study is macroencapsulation by which hazardous waste constituents are physically encapsulated in discontinuous pores within the stabilizing/solidifying materials. However, before being subjected to the leaching test, the solidified products were pulverized by grinding to a particle size smaller than 9.5 mm. This procedure reduced the role of macroencapsulation on waste immobilization because most of the macroencapsulation was supposed to be destroyed. Consequently, the latter mechanism, chem-

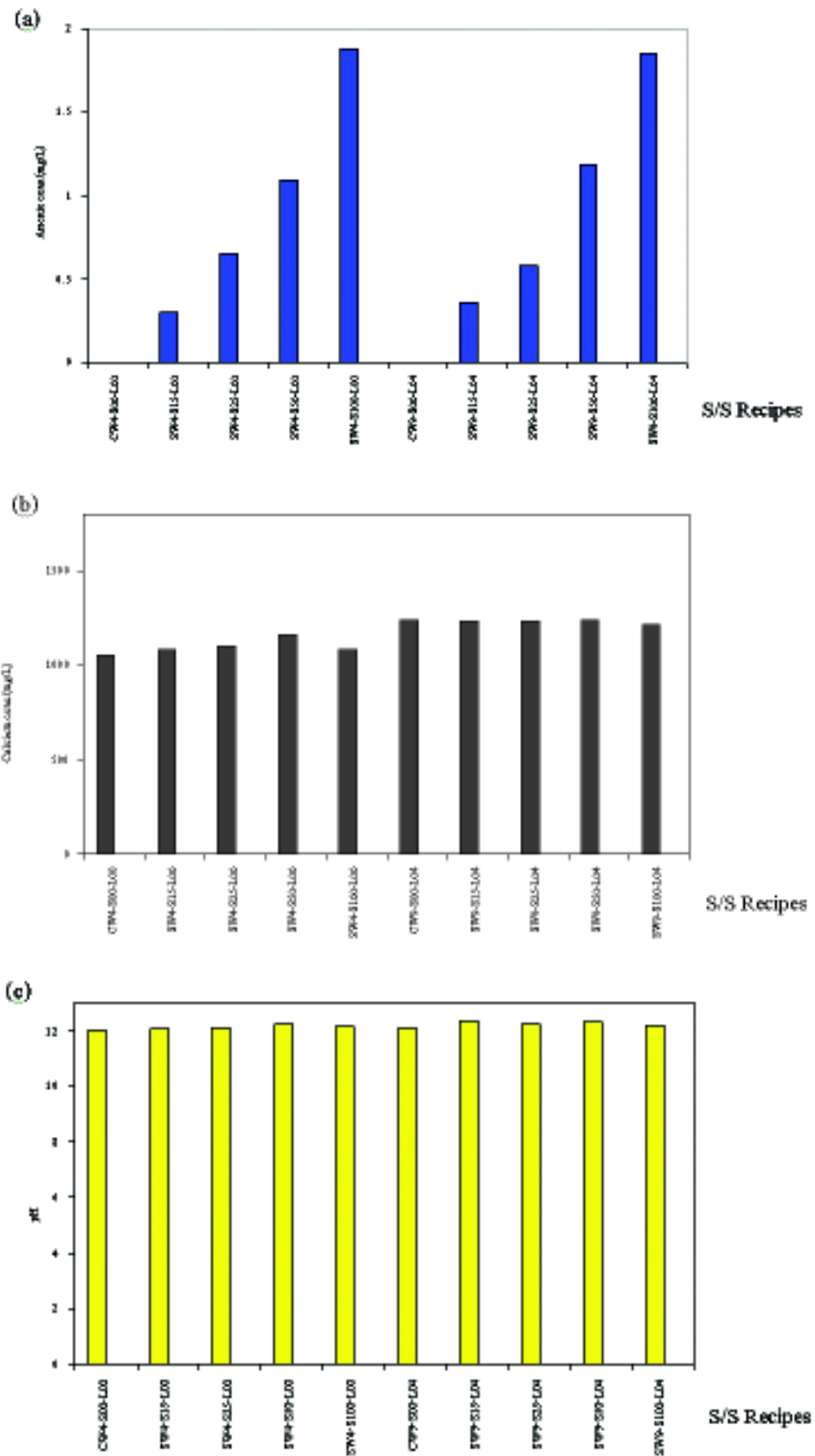


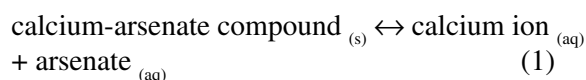
Figure 4. The concentrations (mg/L) of (a) As, (b) Ca, and (c) pH of leachate

ical immobilization, should play the more important role. It is supposed that during the leaching test arsenic desorbed from the sludge reacted with calcium leaching from both calcium hydroxide and C-S-H of the pulverized S/S matrix to form another type of insoluble calcium-arsenic compound.

However, the issue is much more complex than only the chemical reaction between arsenic and calcium discussed in the previous paragraph. Considering Figure 4 (a), it is evident that leaching of arsenic from the solidified/stabilized waste shows direct correlation to the addition of the sludge. This correlation violates the concept of solubility product for metal species in terms of the equilibrium constant governing arsenic immobilization by formation of a calcium-arsenic compound in the leachate of S/S waste mentioned above. Therefore, the second issue focusing on this extraordinary experimental result arises from this point, and the reaction between calcium and the two major species of arsenic found in the sludge, arsenate and arsenite, in the leachate at equilibrium are separately described to obtain the reasonable explanation of this issue.

To understand how this correlation violates the solubility product concept as well as to find the possible explanation for the issue, it is worthwhile to apply the general theory of equilibrium to the formation of a calcium-arsenic compound during the leaching test.

In the leaching test, not only did calcium leach out of $\text{Ca}(\text{OH})_2$ as well as other hydration products but also arsenic desorbed out of the sludge which once was encapsulated in the solidified matrix. Consequently, under equilibrium the reaction between calcium ion and arsenate could be expressed as:

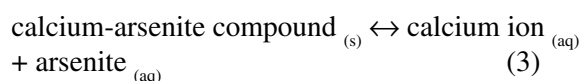


There, the dissociation constant is expressed as:

$$K_d = \frac{[\text{calcium ion}] \times [\text{arsenate}]}{[\text{calcium-arsenate compound}]} \quad (2)$$

Based on MINEQL⁺, the chemical equilibrium modeling system (MINEQL⁺, 1998), at the high concentration of calcium as measured in the present study, arsenate should not be present in aqueous phase but precipitated as calcium arsenate compound.

In the same way, under equilibrium the reaction between calcium ion and arsenite can be expressed as:



There, the dissociation constant is expressed as:

$$K_d = \frac{[\text{calcium ion}] \times [\text{arsenite}]}{[\text{calcium-arsenite compound}]} \quad (4)$$

In the above equation, the concentration of calcium-arsenite compound, which is in solid phase, is constant. Multiplying both sides of equation (4) by [calcium-arsenite compound] results in a different value for K_d which is known as K_{sp} , the solubility product:

$$K_{sp} = [\text{calcium ion}] \times [\text{arsenite}] \quad (5)$$

This equation concludes that, for dissociation of solids such as metal compounds including the arsenite compound, the concentration of metal ion in the leaching solution is not affected by the concentration of the solid metal compound in the waste.

According to recent study (Vandecasteele *et al.*, 2002; Palfy *et al.*, 1999; and Dutré and Vandecasteele, 1998), the possible form of calcium-arsenite compound is CaHAsO_3 of which K_{sp} , without consideration of activity coefficients, is 1.07×10^{-7} . For this reason, the matter of that increased amount of the sludge addition may result in increasing potential for arsenite leaching

out during the extraction test should have not been the main factor controlling arsenite concentration in leachate, but the real controlling factor should have been calcium ion. Based on equation (5), concentration of arsenite in each S/S recipe can be approximately calculated as :

$$[\text{arsenite}] = \frac{1.07 \times 10^{-7}}{[\text{calcium ion}]} \quad (6)$$

By this equation, at the $[\text{Ca}^{2+}]$ around 1171.33 mg/L, the average calcium concentration of all leachate in the present study, arsenic concentration should have been only around 0.27 mg/L regardless of the amount of the sludge added. Therefore, the difference between the calculated value and the observed values suggests that there must be another factor beside the formation of CaHAsO_3 controlling immobility or mobility of arsenic in leachate.

This governing factor may be pH. By considering formation of CaHAsO_3 , it is found that the presence of Ca^{2+} and HAsO_3^{-2} is required to form this compound, and the factor determining how many percent of total arsenite ion present in form of HAsO_3^{-2} is pH. Calculated by MINEQL⁺, it is found that at around pH 12, the average pH of all leachates in this study, only around 43.88 % of total arsenite is in the form of HAsO_3^{-2} which means that other 56.12 % is in the forms which cannot form CaHAsO_3 . By this reason, it is not surprising why there is a close relationship between the increasing amount of sludge and the increasing concentration of arsenic in leachate.

3. Evaluation of utilization potential

Generally speaking, there are two matters of concern regarding utilization of a solidified waste form as construction materials. First, the solidified product must not pose any threat to the environment during utilization. The arsenic leachability of the solidified products following LP-No.6 can be used as a rough indicator to address this issue due to the fact that according to this leaching procedure the macroencapsulation of

solidified products is destroyed, thus the simulation is believed to represent the worst-case scenario. Based on this criterion, the utilization of the solidified waste form is supposed to pose no unacceptable threat to the environment because of the fact that, as shown in Figure 4, the concentrations of arsenic in the leachate pass the regulatory limit with a wide margin.

The second issue is the physical requirement in terms of strength of the matrices. According to TIS 827-2531 (TISI Standard, 2003), the required unconfined compressive strength of a product to serve as an interlocking concrete paving block is around 410 ksc. Consequently, as illustrated in Figure 1, the solidified product with waste-to-binder ratio of 0.15 without addition of lime has the potential to be utilized as an interlocking concrete paving block. However, it should be noted that the present study serve to provide a preliminary information on the utilization potential of the solidified/stabilized waste form. In order to reach the point of practical application, the product with the actual size and shape, has to be subjected to many other tests according to the TIS 827-2531 (TISI Standard, 2003).

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